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AGING OF CURE DATED ITEMS
AND VARIOUS ELASTOMERIC
COMPOUNDS

To: Oklahoma City Air Materiel Area
Tinker Air Force Base, Oklahoma

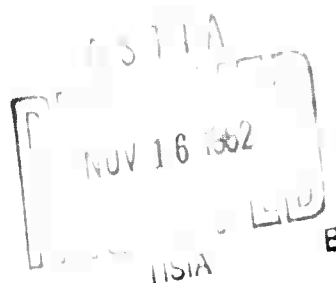
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Final Report
Contract AF 34(601)-5233PA
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INTRODUCTION

The following text represents a summary of the work performed on Project 1190-18 at the University of Oklahoma Research Institute under Contract AF 34(601)-5233PA for the Oklahoma City Air Materiel Area. The report is entitled: Aging of Cure Date Items and Various Elastomeric Compounds.

The report is divided into eleven separate sections. Each section describes a particular phase of the entire problem that was studied and evaluated. Conclusions have been made wherever possible.

SECTION I

**Correlations Between Natural and Accelerated
Aging and Mechanical Properties**

An extensive literature search has been carried out in order to obtain information relative to the air aging of nitrile rubber. It was desired to obtain a correlation between degradation (aging) and a corresponding change in engineering properties. Results of testing programs indicate that tensile strength and modulus may increase, decrease, or remain essentially unchanged while ultimate elongation decreases during aging for all types of rubber. The elongation decrease seems to vary regularly with time, thus enabling extrapolation of high temperature tests to room temperature shelf aging in some cases.

Air Oven Method

Tests have been conducted to determine the best method of accelerated high temperature aging: the air oven, oxygen bomb, and air bomb were compared. ⁽¹⁾ The air oven method gave more reproducible and uniform results than the oxygen and air bomb method. Work discussed in this report was performed by the air oven method.

Some difficulty arises in the air oven method because of the loosely defined operating conditions. One particular and very significant variable is air circulation. If correlation of data from different laboratories is desired the amount of air circulation must be controlled very precisely. A method that seems to improve the air oven technique is the test tube method, where samples are placed in a sealed test tube and inserted in the air oven.

Discussion

The physical changes in nitrile rubber vulcanizates that occur between 150°C and room temperature seem to be the same and have an

activation energy of 19,000± 2000 cal/mole; (2) therefore, extrapolation from high temperatures to room temperature should be permissible. But extrapolation is possible only if: 1) accelerated aging temperatures and service temperature are in the range of the same degradation mechanism, that is, degradation varies with time in a regular way; and, 2) the temperature dependence of the rate-limiting reaction for the specific rubber is known; that is, the actual rate that the physical properties vary with time-- some empirical equation to relate time with temperature.

Juve and Schoch have made extensive tests on aging of nitrile and other types of rubber (2,3,4). In the first and second publications tests were reported at temperatures of 121°C, 100°C, 70°C, 25°C and an activation energy calculated. The data was somewhat incomplete and scattered but a linear correlation was shown when the reciprocal absolute temperature was plotted versus \ln (time) at a constant loss of elongation. Hence, extrapolation to room temperature was possible. This is shown in Figure 1.

During the same period a 12 year testing program was being carried out in the United States (75°F) and in Liberia (85°F). Results of this test are shown in Table I and the data plotted in Figure 2.

FIGURE 1

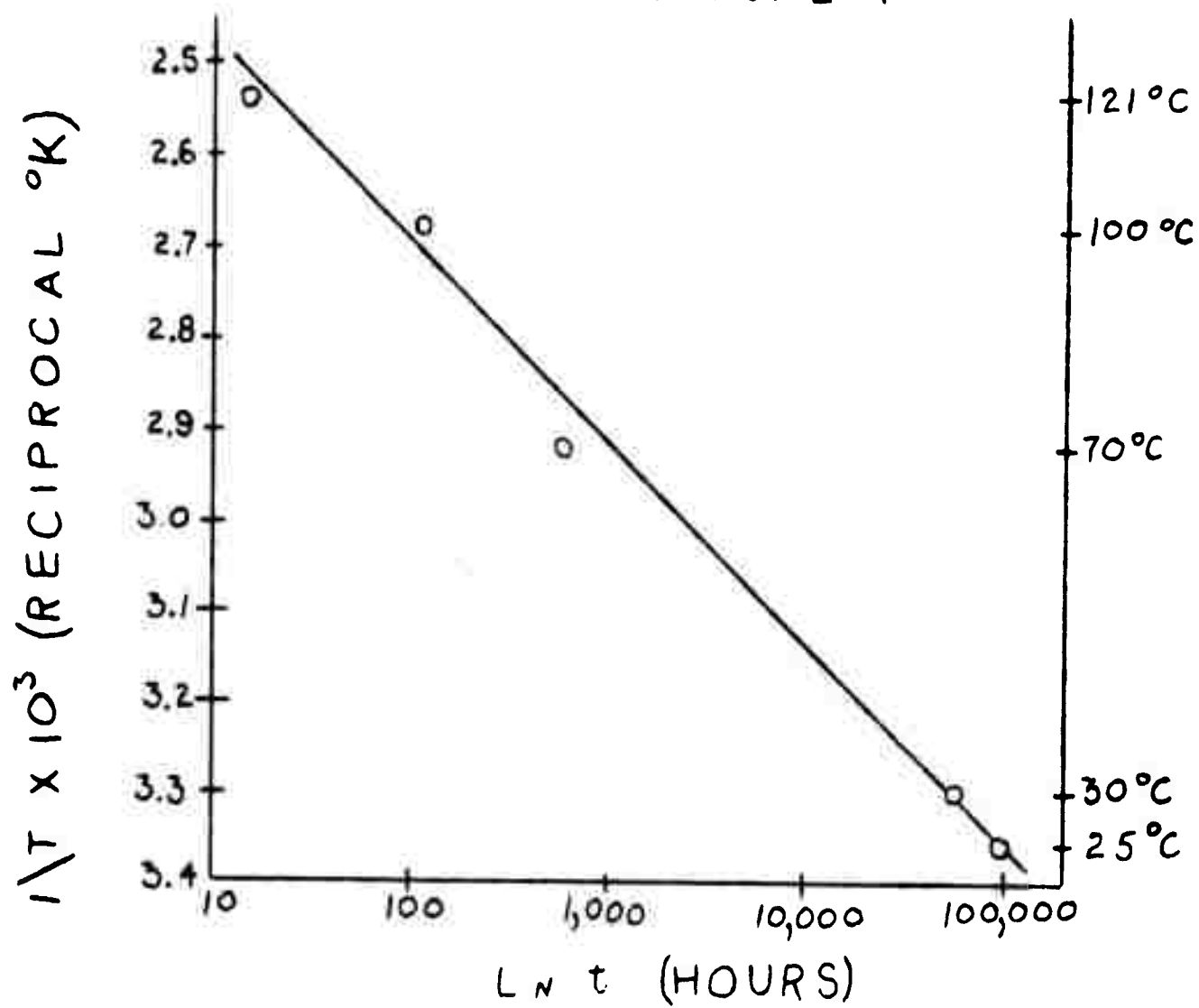
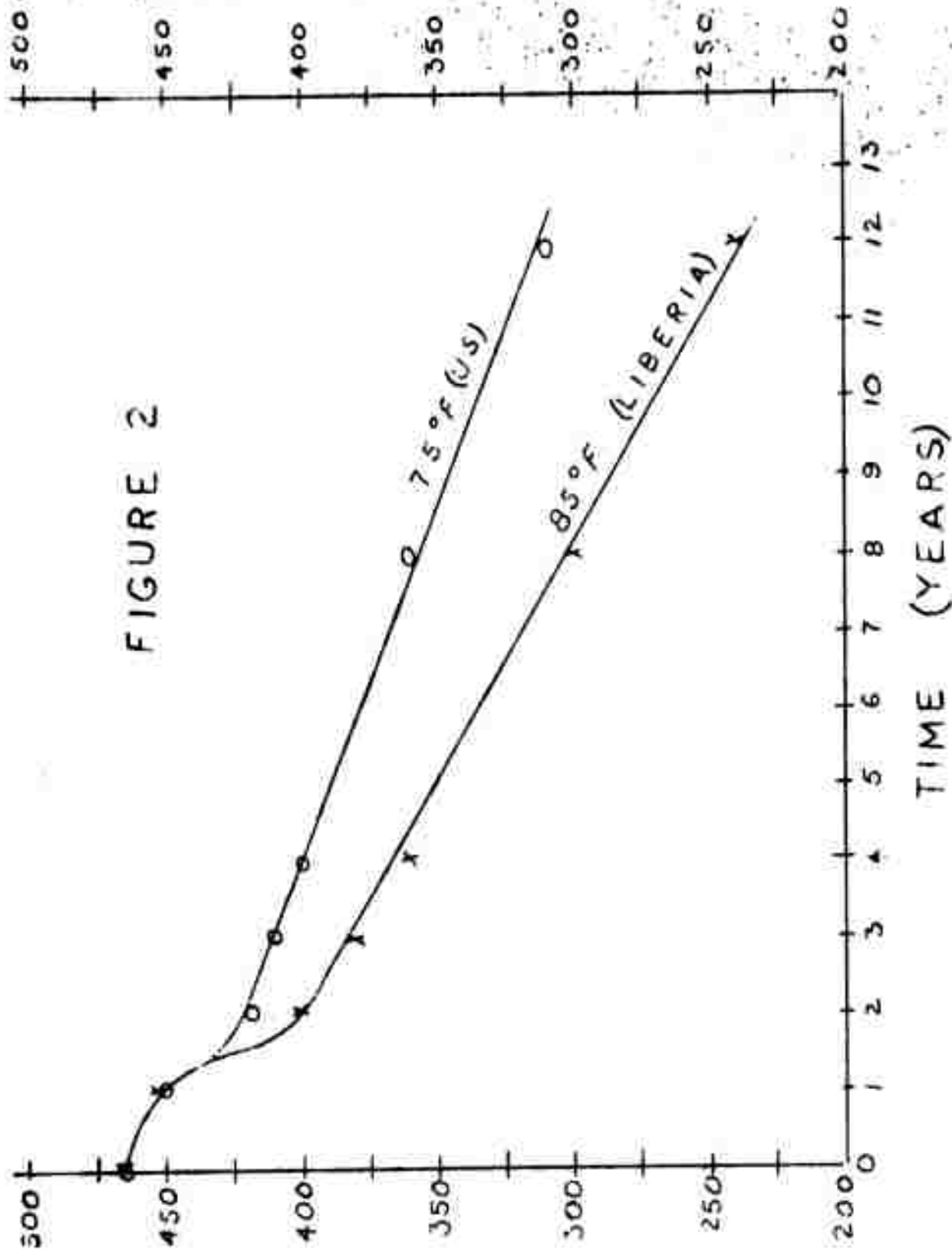


TABLE I

90' at 275°F Cure

<u>Aging Time</u>	<u>300% Mod.</u>	<u>% Retained</u>	<u>Tensile Strength</u>	<u>% Retained</u>	<u>Elong.</u>	<u>% Retained</u>
Original	1745	100	2500	100	465	100
1 year U.S.	1816	104	2487	99.5	450	96.7
2 years U.S.	1943	111.2	2450	98	417	89.6
3 years U.S.	1950	112	2540	101.6	410	88.1
4 years U.S.	2080	119.2	2580	103.1	400	86
8 years U.S.	2255	129	2545	102	362	77.8
12 years U.S.	2460	141	2700	108	310	66.6
1 year Liberia	2275	130.2	3210(?)	128.5	453	97.3
2 years Liberia	2100	120.2	2625	105	400	86
3 years Liberia	2200	126	2525	101	380	81.6
4 years Liberia	2400	137.5	2725	109	360	77.3
8 years Liberia	2625	150.5	2625	105	300	64.4
12 years Liberia	-	-	2575	103	240	51.6

ULTIMATE ELONGATION (PERCENT)



Linear correlation is possible and yields a 33.4% decrease in elongation in 12 years. This decrease in elongation is permissible in most applications.

Mandel et al. (5) of the National Bureau of Standards made room temperature and high temperature tests independently and also used the data from the test previously mentioned and attempted to extrapolate to room temperature. It is important to note that different recipes were used.

TABLE II

Juve and Schoch (2)

Nycar OR-15	100 parts
Zinc Oxide	5
Stearic Acid	1
Sulfur	1.5
Benzothiazyl disulfide	1.5
SRF Black	75
Plasticizer SC	15
TEP	15

Mandel et al. (5)

Nitrile Rubber	100 parts
Zinc Oxide	5
Stearic Acid	1.0
Sulfur	1.5
Benzothiazyl disulfide	(?)
Gas Furnace Black	40

Mandel found that the equation $E = E_0 - kt^{\frac{1}{2}}$ satisfactorily represented the behavior of nitrile rubber. E is the elongation after aging, E_0 and k are constants for a specific type rubber, and t the time in days. The change in elongation, $E - E^*$, is plotted with respect to $t^{\frac{1}{2}}$ at each test temperature (E^* is the initial elongation). The

values of k and E_0 are read off the plot as the slope and ordinate intercept respectively. Results of Mandel's study of accelerated aging are shown in Figure 3. Correlation of data from Juve and Schoch by the equation proposed by Mandel is shown in Figure 4.

Values of k were determined at the measured temperatures and are listed in Table III.

TABLE III

<u>Juve and Schoch</u>		<u>Mandel</u>	
<u>Temp °C</u>	<u>k</u>	<u>Temp °C</u>	<u>k</u>
25	2.76	23	6.4
70	47	34	8.9
100	75	45	11.4
121	154	57	21.1
		70	33.6
		85	74.2
		100	180.4

An attempt was made to correlate $\ln k$ with reciprocal temperature in hopes that a linear correlation may be obtained. Figure 5 shows this correlation. The non-linear plot from Mandel's work is in direct disagreement with the work of Juve and Schoch and says effectively that room temperature aging can not be predicted from high temperature results only. Mandel reports that by using the above method the aging time for most rubber compounds may be predicted up to 10 years, but that the values of k and E_0 must be experimentally determined for each type rubber.

Analysis

Only two sources of data on nitrile rubber aging were found in the literature, and the two sets of data do not agree. Mandel's analysis

ULTIMATE ELONGATION (PERCENT)

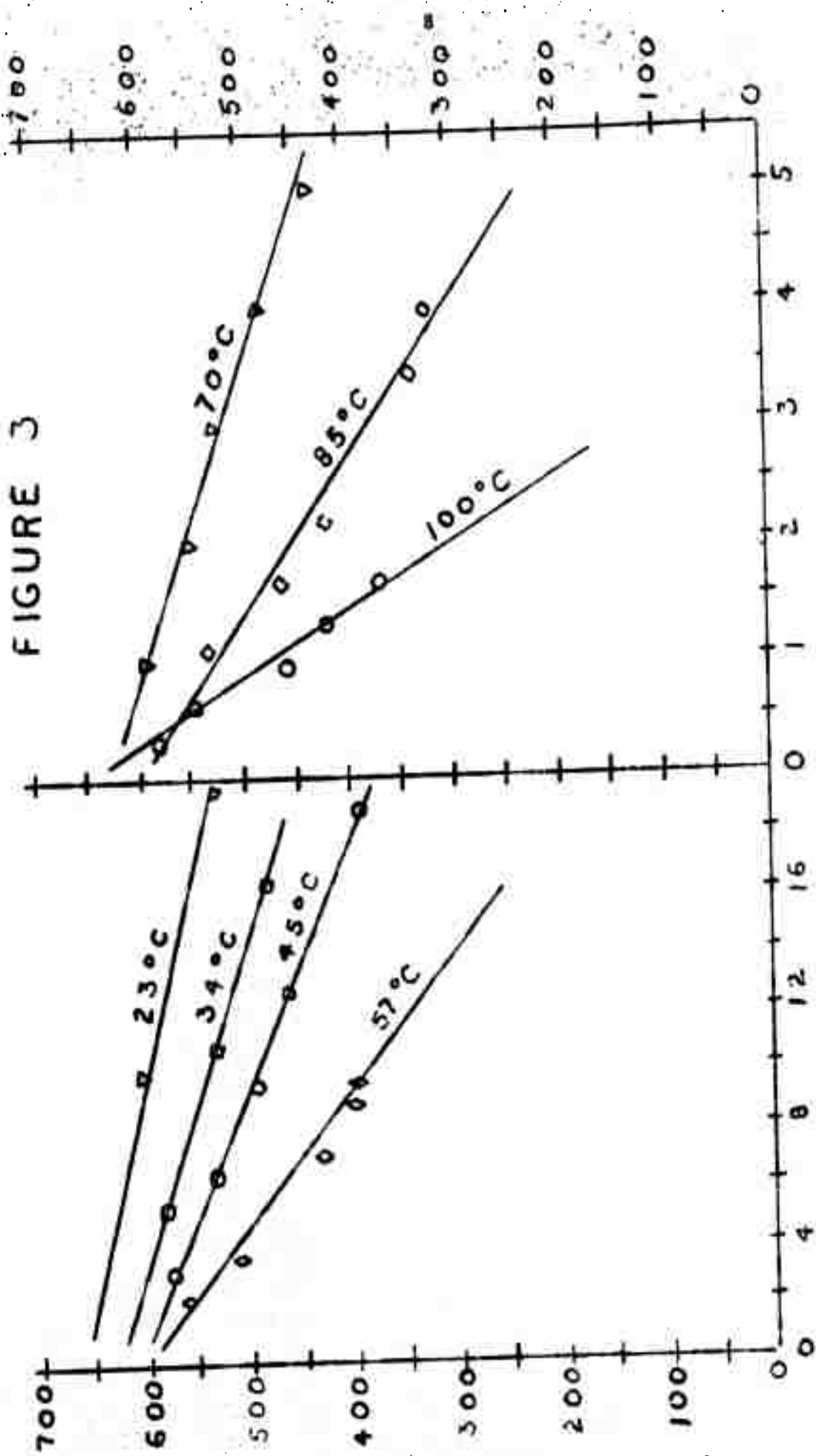


FIGURE 3

$[TIME]^{1/2}$ (DAYS)

ULTIMATE ELONGATION (PERCENT)

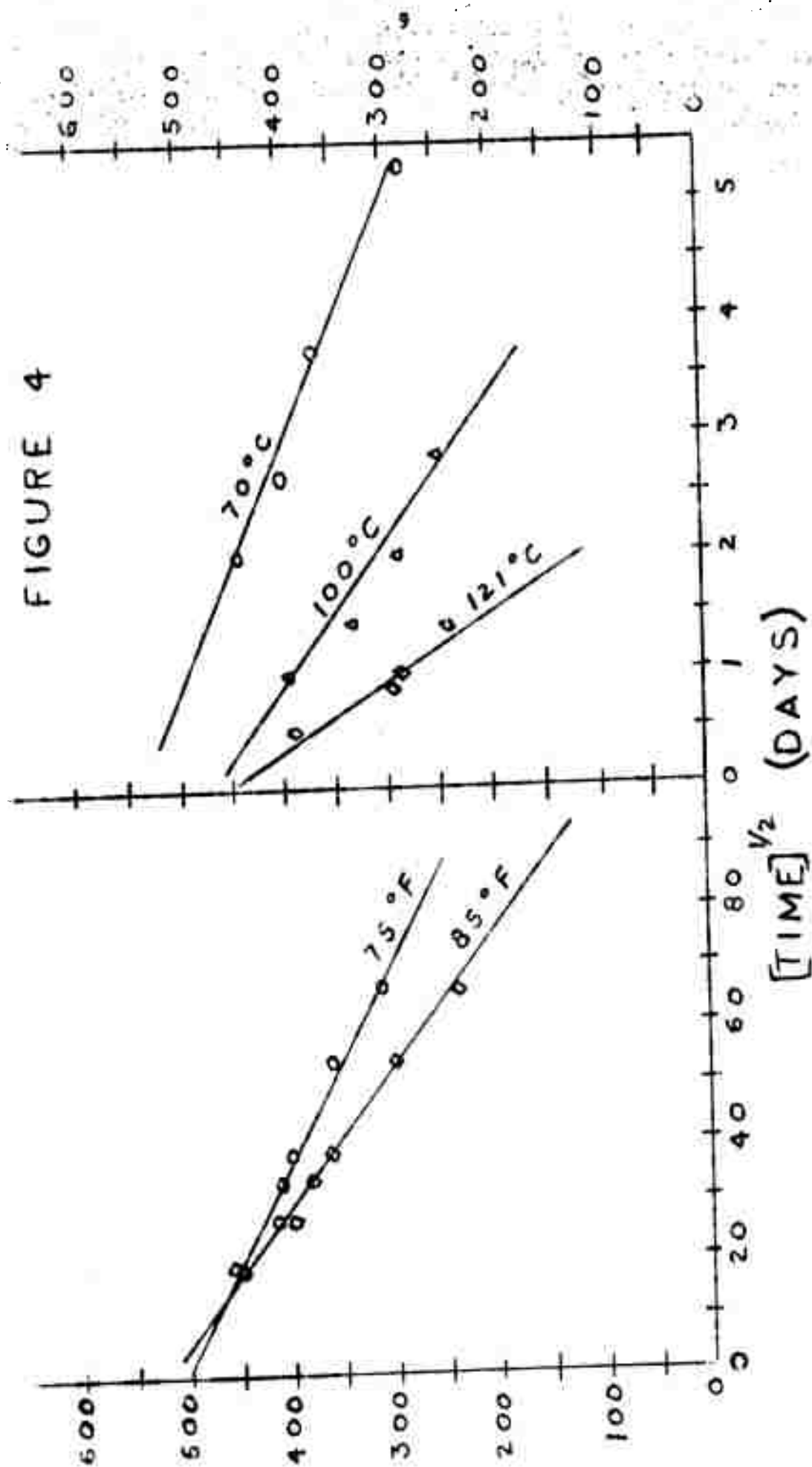
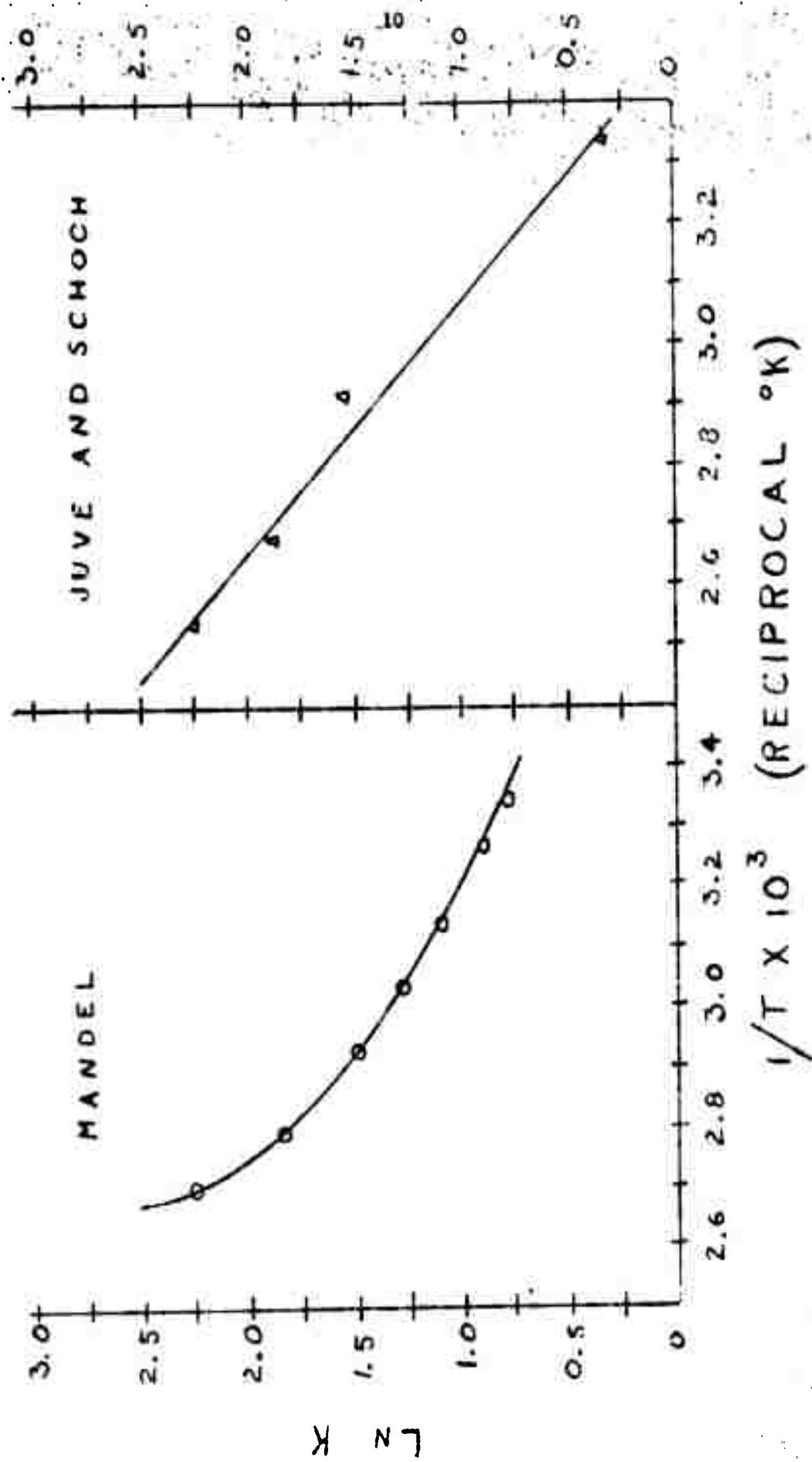


FIGURE 5



of the non-linear variation of k with $1/T$ seems to be consistent and represents a more sound approach than that of Juve and Schoch.

Figure 6 shows the analysis of data with the equation, $E = E_0 - kt^{\frac{1}{2}}$. The triangle points represent calculated data with an elongation decrease of 33.4% and the circles a decrease of 14%. The values for elongation decrease are taken from the 12 year in situ test discussed earlier. Points on the figure were plotted from calculations utilizing determined k -values and a 33.4 and 14% elongation decrease at a particular temperature. Because of the limited extent of data available it was possible to draw several lines through the data points. The data at 25°C was calculated from short time tests and extrapolated. A line through the four calculated points comes fairly close to the measured values (the 70°C point seems bad in both cases), but using only the high temperature point results in gross error. A predicted value for 14% decrease in elongation is 2.4 years (4 years actual) and for a 33.4% decrease-- 13.3 years (12 years actual).

It is of interest to note that elongation is not a linear function in the early stages of deterioration (Figure 2). A similar trend was observed in preliminary tests made at OCAMA. The tests were discontinued after 91 days but the non-linear portion of the curve is evident and the correlation is shown in Figure 7.

FIGURE 6

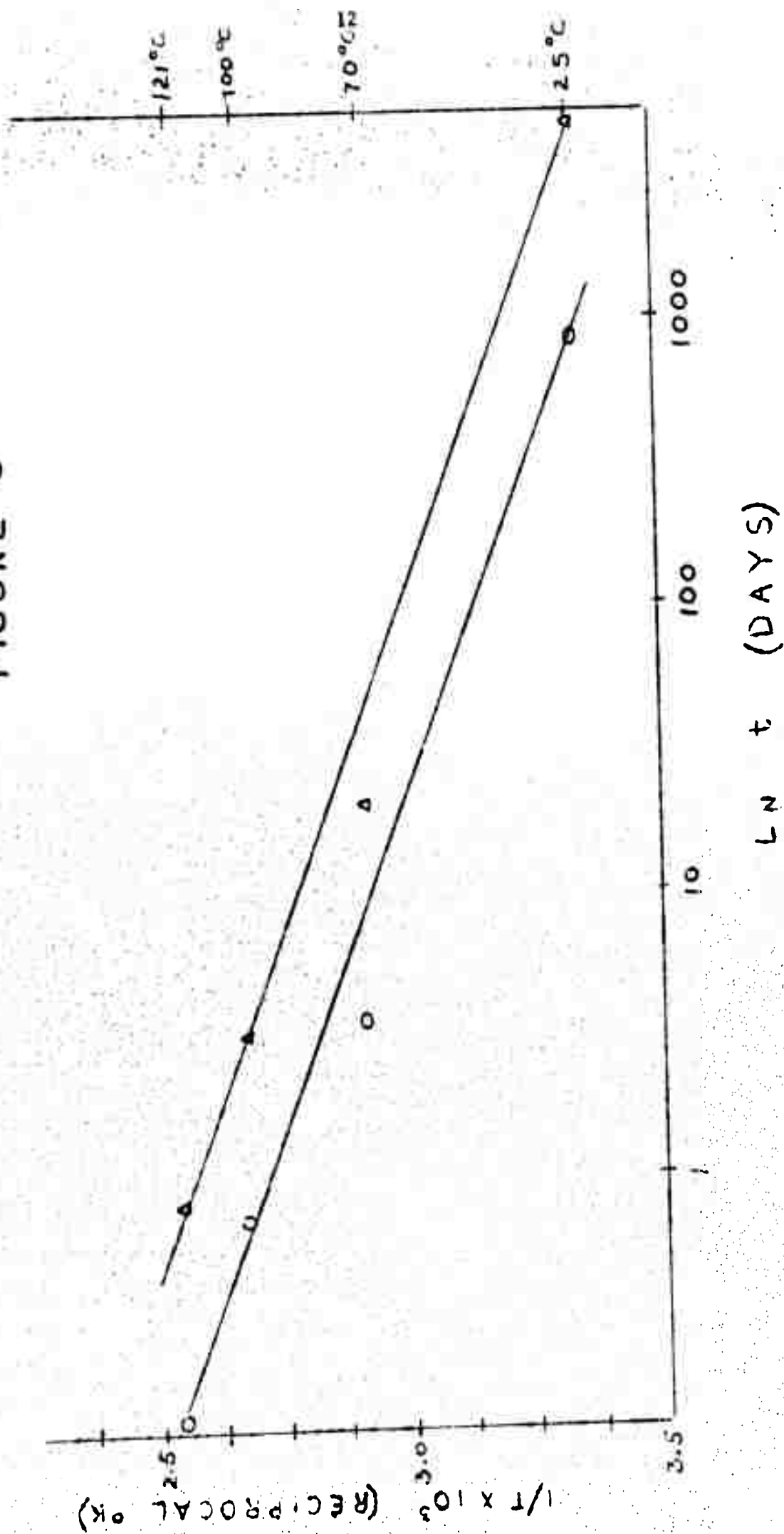
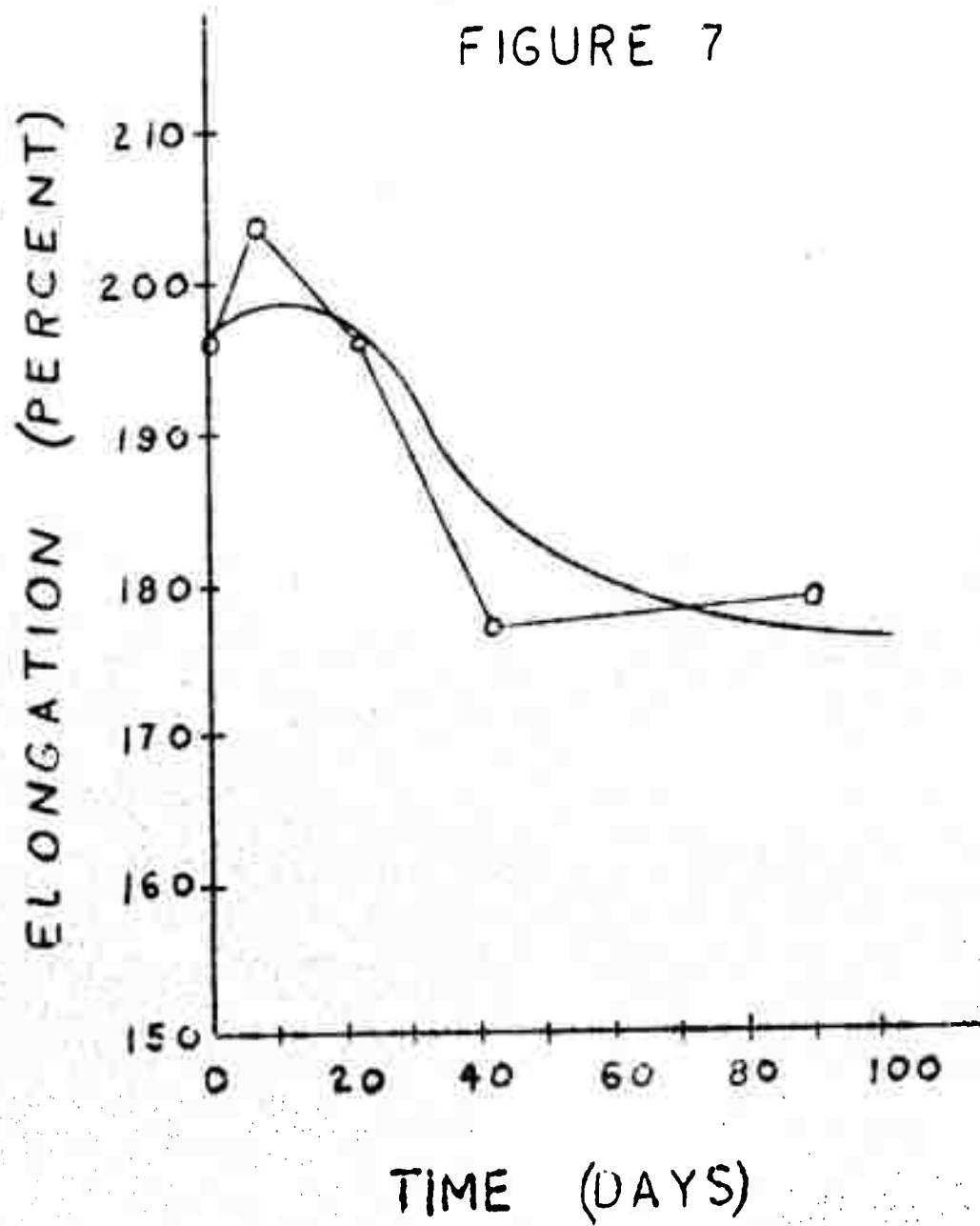


FIGURE 7



OCAMA TESTS AT 70°C

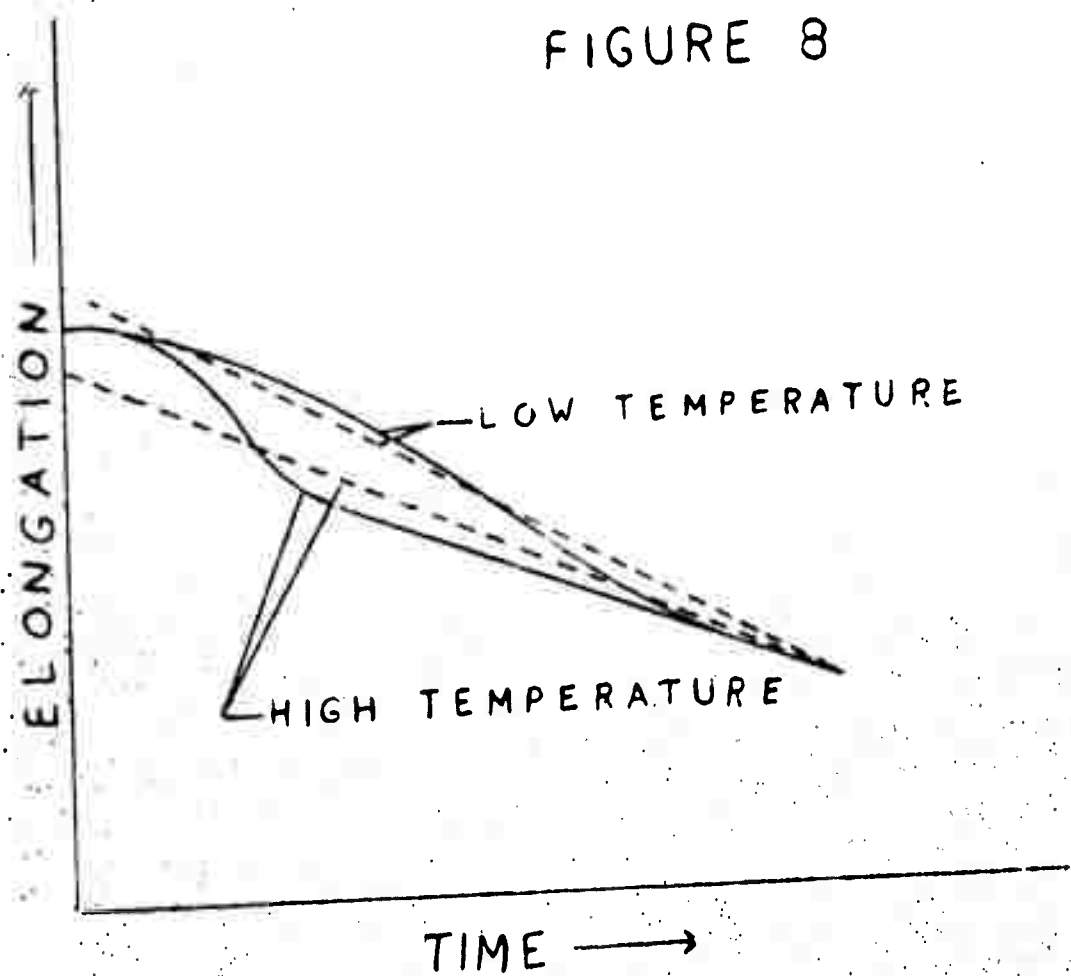
TABLE IV

	<u>Temp.</u> <u>(°C)</u>	<u>E*</u> <u>(Percent)</u>	<u>E₀</u> <u>(Percent)</u>	<u>E₀ - E*</u> <u>(Percent)</u>
Mandel, et al.	23	652	657	+ 5
	34	623	626	+ 3
	45	609	608	- 1
	57	614	603	-11
	70	596	613	+17
	85	609	600	- 9
	100	656	647	- 8
		<654>		
Juve and Schoch	25	465	500	+35
	70	465	532	+67
	100	465	468	+ 3
	121	465	450	-15

In Table IV values are given for E^* (initial elongation) and E_0 at different temperatures from two different experiments (2,5). As can be seen the value of $E_0 - E^*$ goes from positive at low temperatures to negative at high temperatures. The relative time duration of the after-vulcanization effect is probably much shorter at high temperature than at low temperature. Then, if two samples of the same rubber were aged at different temperatures for a short time, the after-vulcanization effect could still be present in the low temperature aging whereas it would have been completed in the high temperature test.

In Figure 8 an attempt has been made to graphically explain the variation of E_0 with E^* . Percent elongation is plotted schematically at two temperatures as a function of time on different time scales for a given loss of elongation. Straight lines are fitted to the data. Note that the low temperature data will give an extrapolated value of E_0 at

FIGURE 8



time zero that is greater than E^* . At higher temperatures the extrapolated value of E_0 is lower than E^* . Thus the value of E_0 and k are functions of the after-vulcanization effect as well as temperature and composition.

Future Work

In all the experimental accelerated aging tests the first determination gave a substantial loss in elongation. It is proposed to make tests at high temperatures, in order to measure significant changes in elongation in short intervals of time. For example, at 100°C determine the elongation every 3 - 4 hours and at 125°C at intervals of 1 - 2 hours.

The change in cross-linking in the samples would be checked by swelling tests to determine how the cross-linking changed with the after-vulcanization effect. The amount of free sulfur could be checked at each interval to determine how much of the free sulfur has reacted. Infrared absorption spectra (7) would possibly indicate changes in chemical groups present and molecular rearrangements.

If the after-vulcanization effect could be understood and compensated, extrapolation of high temperature accelerated aging data to shelf aging temperatures would be more reliable than at the present time.

SECTION II

Vapor Phase Swelling

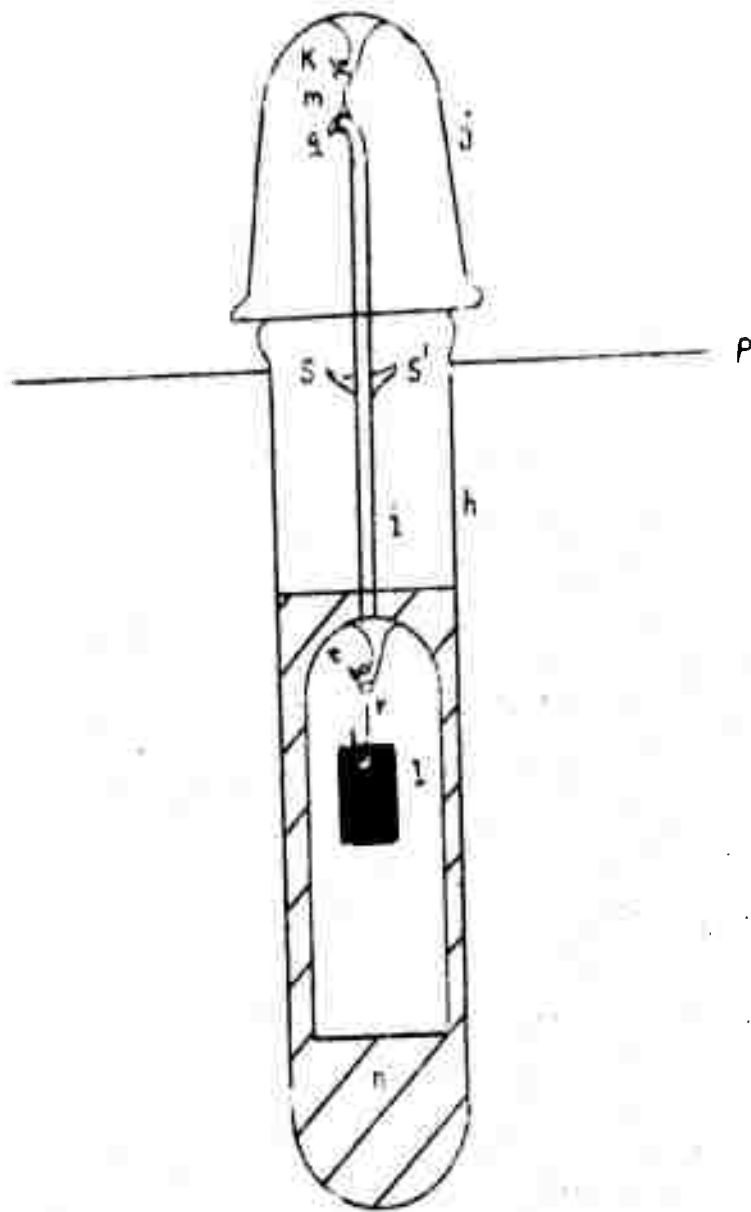
Introduction

In order to study the swelling of elastomers in organic solvents a new technique was devised that would allow determination of the extent of swelling as a function of varying solvent activity or partial pressure. The advantage of this technique over the more common techniques is that swelling measurements can be made without the necessity of immersing the sample in the swelling liquid. This not only eliminates leaching of soluble materials from the rubber but permits swelling studies at any desired solvent vapor pressure instead of only the saturation vapor pressure to which the immersion techniques are limited.

Apparatus

The modified test tube apparatus devised for this technique is shown in Figure 9. Tube h is an 8 inch test tube fitted with a 24/40 f cap j, which has a small glass hook, k, sealed to the inside of the top. A glass insert i is suspended from the hook, k, by means of a wire, n, and hook q. The lower portion of the insert has a hook, t, sealed to the inside and is completely surrounded by the swelling fluid, m. The rubber sample, l, is suspended from hook t by means of a wire, r, about one cm. or so below hook t. When the insert is lowered into the swelling fluid trapped air prevents the fluid from rising into the portion of the insert containing the rubber sample. To prevent the insert from floating in the fluid lead collars were placed around the stem of the insert. Hooks s and s' prevent the collar from slipping to the lower portion of the stem and can also be used to suspend additional weights if necessary. The entire tube is wrapped in aluminum foil to

FIGURE 9

MODIFIED TEST TUBE
SWELLING APPARATUS

aid in maintaining constant temperature and the tube is immersed in constant temperature bath p , so that the surface of the swelling liquid is several inches below the water surface. Several samples are run simultaneously by supporting six such tubes in the same constant temperature bath.

The swelling fluids used consisted of mixtures of hexadecane and benzene for which total vapor pressure had been determined as a function of the concentration. Approximately 30 ml. of varying concentrations of this mixture was placed in each tube.

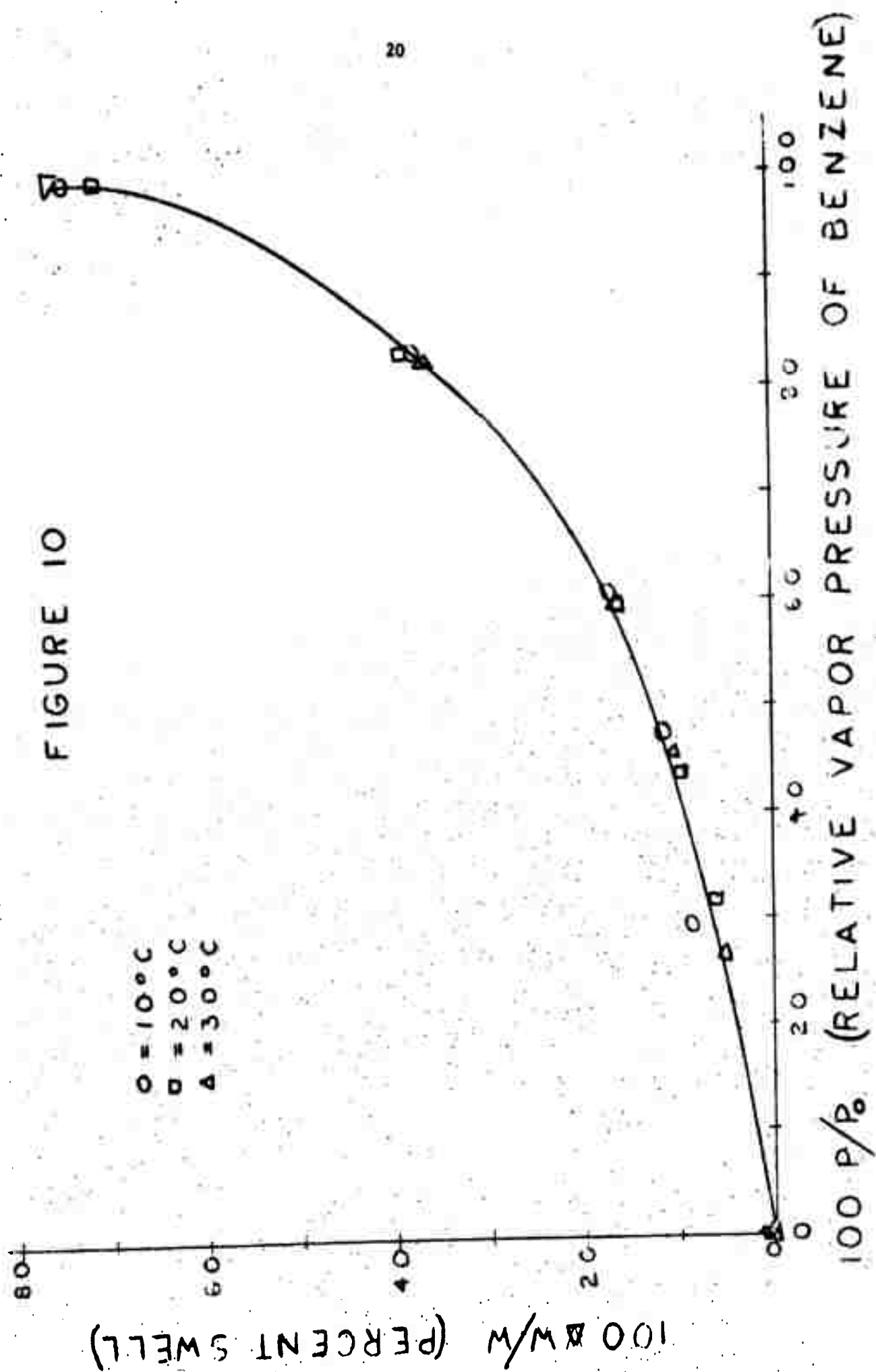
Procedure

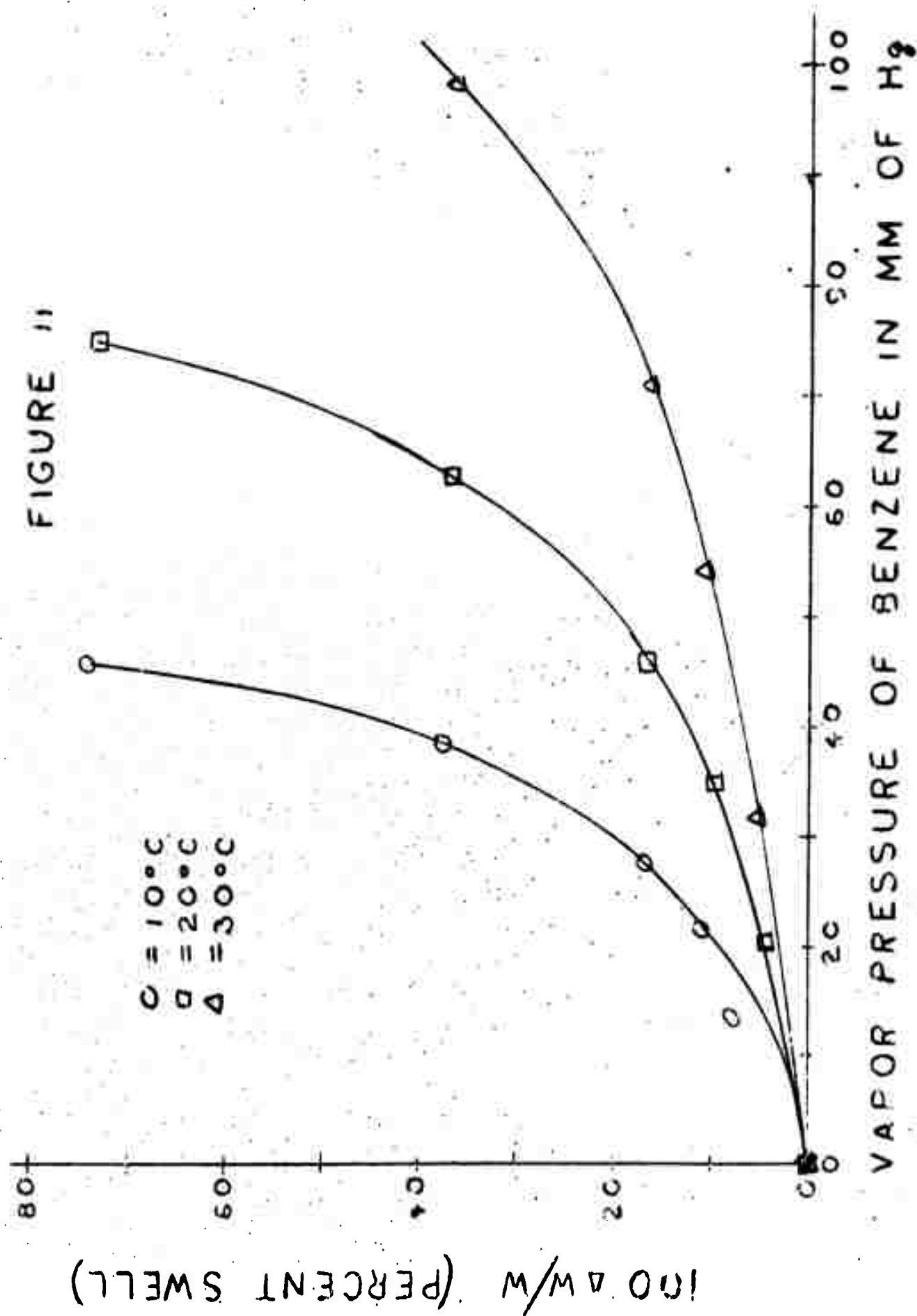
In making a run, weighed samples were suspended in the apparatus and the constant temperature bath was adjusted to the required temperature. After equilibrating for several days, each sample was taken from the apparatus, quickly transferred into a pre-weighed weighing bottle, and weighed. The concentration of benzene in the swelling fluid was determined by a refractometer measurement. It was discovered that samples reached a nearly constant weight within 2 or 3 days; hence, as a standard procedure, each specimen was left in the apparatus for three days.

Preliminary Results

Swelling isotherms were determined for specimens of a Buna-N type rubber. The samples used in the modified test tube method measured approximately 1 cm X 2 cm X 3 mm, and weighed between 0.5 and 1.0 gram.

Results of measurements on the Buna-N samples in benzene are given in Figures 10 and 11, where isotherms at 10° , 20° , and 30°C are shown.





In Figure 10 percent weight increase, $(\Delta W/W)100$, is plotted versus percent of saturation vapor pressure, $(P/P_0)100$. In Figure 11 percent weight increase, $(\Delta W/W)100$, is plotted versus vapor pressure of benzene.

In order to illustrate the advantage of the test tube swelling method over the immersion techniques, six rubber samples of the type used in the swelling determinations were immersed in benzene which was contained in an enclosed tube to prevent evaporation and allowed to swell from 2 to 118 hours. At a prearranged time the sample was removed from the solvent and evacuated under reduced pressure in a vacuum desiccator until a constant residue weight was obtained. The weight percent of the sample dissolved by the solvent was calculated by dividing the residue weight by the original sample weight. At the same time another sample was suspended above pure benzene for 120 hours. The benzene vapors were absorbed by the sample until the sample had become saturated, at which time the vapors condensed on the surface of the sample and dripped back into the liquid solvent producing a slight leaching effect.

In the tubes containing the immersed samples the sample became saturated with solvent within the first ten hours (The time required for saturation had been determined in a previous experiment). Leaching of the sample occurred rapidly during this time and appeared to become non-existent after the sample became saturated. However, more than five percent of the sample had been dissolved at the end of two hours and more than ten percent had been dissolved at the end of 22 hours. On the other hand the sample suspended above the solvent became saturated with solvent within 72 hours but at the end of 120 hours had lost less

then one percent of its weight due to dissolution. Results of this experiment are summarized in Table V.

TABLE V

<u>Initial Sample Weight (grams)</u>	<u>Weight of Sample Dissolved (grams)</u>	<u>Type of Solvent Exposure</u>	<u>Time of Solvent Exposure (hours)</u>	<u>Percent of Sample Dissolved</u>
0.8350	0.0425	Immersion	2.0	5.09
0.8129	0.0729	Immersion	7.5	8.97
0.8047	0.0857	Immersion	22.5	10.65
0.8456	0.0827	Immersion	46.5	9.78
0.7983	0.0809	Immersion	67.0	10.13
0.8713	0.0872	Immersion	118.0	10.01
0.7520	0.0062	Suspension	120.0	0.82

Vapor Swelling Studies

Vapor swelling studies were conducted on two Buna-M type rubbers (BN-81848-R from Continental Rubber Works and SR-822-60 from Stillman Rubber Company) and one silicone rubber to determine if there was a correlation between the degree of loss of useful properties of the rubber and its swelling behavior.

The first phase of this study involved the determination of the change in the swelling ability of a rubber sample with prolonged exposure to moderately high temperatures. The rubber samples were heated in a drying oven for the desired length of time, removed from the oven and allowed to cool to room temperature overnight. The samples were then weighed, swelled in the modified test tube apparatus for six days at 20°C and approximately 65.6 mm of benzene vapor pressure (normal vapor pressure of benzene at 20°C is 75.1 mm.), and reweighed to determine the amount of vapor absorbed by the rubber.

The results of one study using all three types of rubber are given in Figure 12, where the percentage change in sample weight due to swelling $[(W/W)100]$ is plotted versus aging time in days. The samples were heated in air at 70°C.

The curves for all three types of rubber show a fairly rapid decrease in swelling ability during the first two weeks followed by a linear decrease for longer periods.

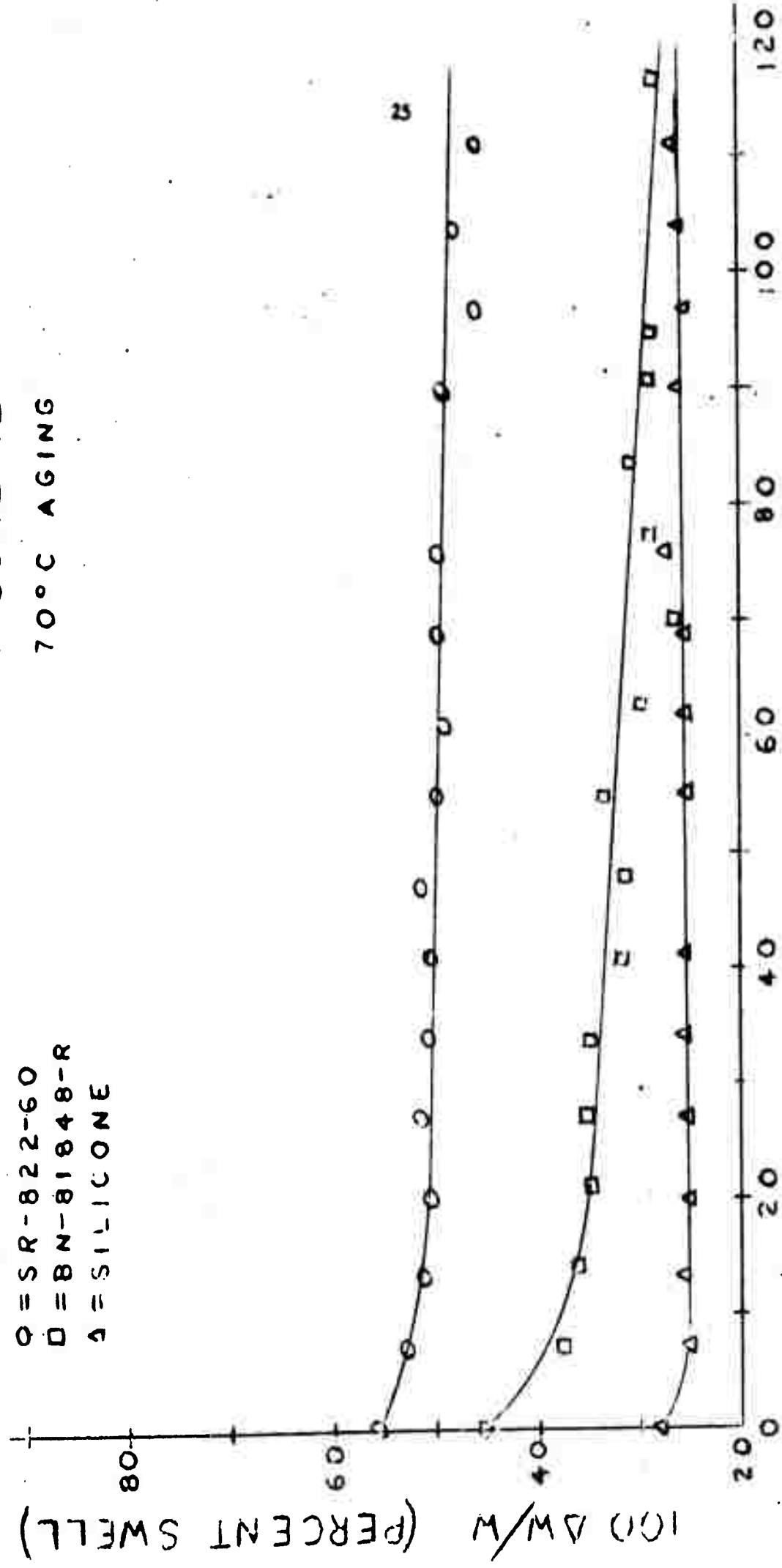
Of the two Buna-N rubbers the SR-822-60 exhibits the greatest stability against aging, while the silicone rubber is the most stable of the three. However, the curves for the two Buna-N rubbers have negative slopes in the linear portions of the curves whereas the linear portion of the curve for the silicone rubber seems to show a very slight positive slope, indicating that its swelling ability improves with aging time. This would in turn indicate a softening of the rubber due to chemical changes occurring in the rubber or to breaking of cross-links in the rubber.

In order to determine the dependence of swelling ability of rubber samples with aging temperature, SR-822-60 rubber samples were heated in air at 70°C and 50°C. After aging for a specified time the samples were removed from the ovens and swelled in the manner described above. Figures 13 and 14 show the results of these tests. Figure 13 is the curve for samples aged at 70°C and Figure 14 is the curve for samples aged at 50°C.

Examination of the curves shows that the rapid decrease in swelling ability during the first two weeks is absent. However, comparison

FIGURE 12
70°C AGING

○ = SR-822-60
□ = BN-81848-R
△ = SILICONE



AGING TIME (DAYS)

FIGURE 13

SR-822-60 SAMPLES
70°C AGING

100 $\Delta W/W$ (PERCENT SWELL)

AGING TIME (DAYS)

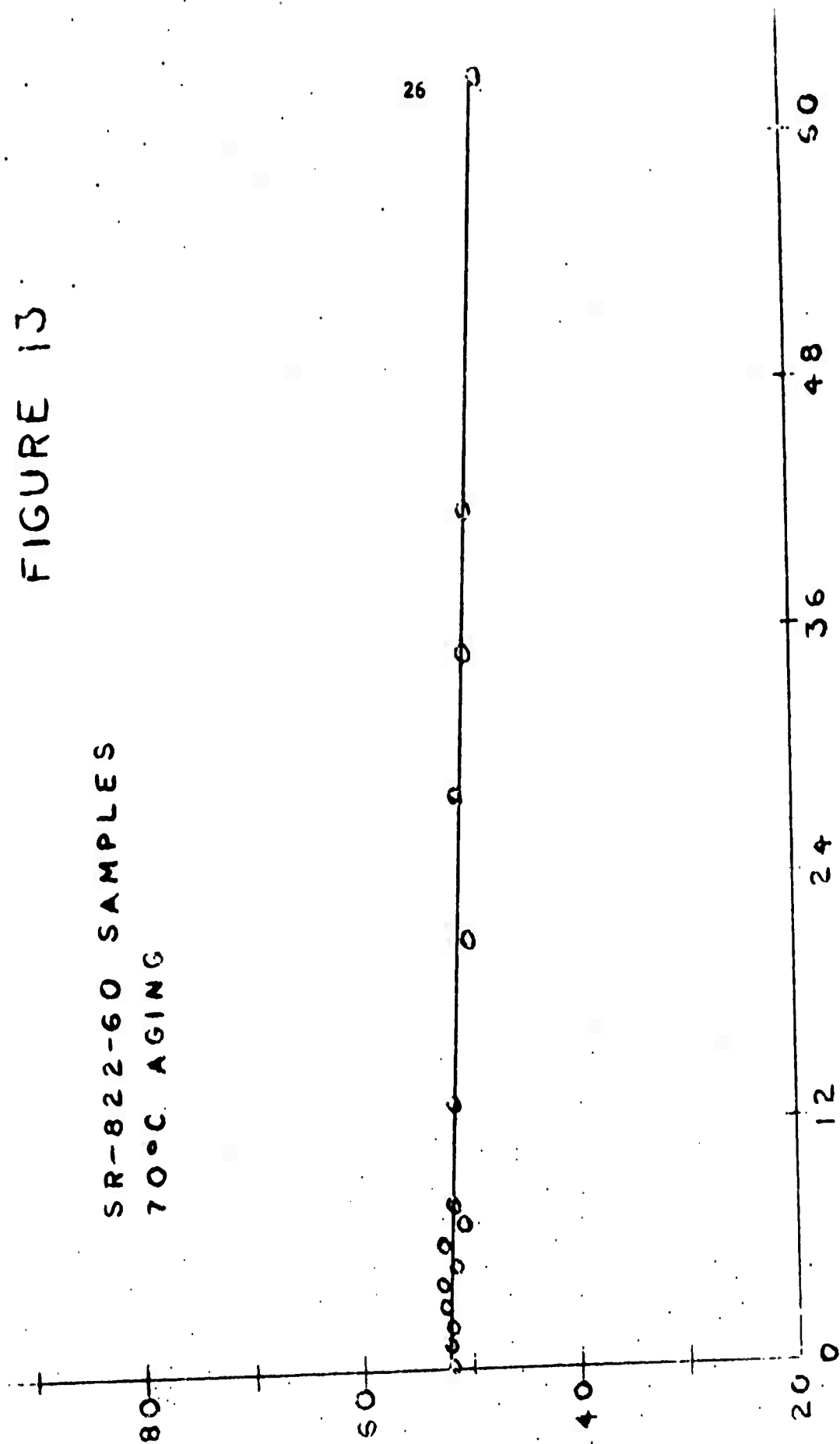
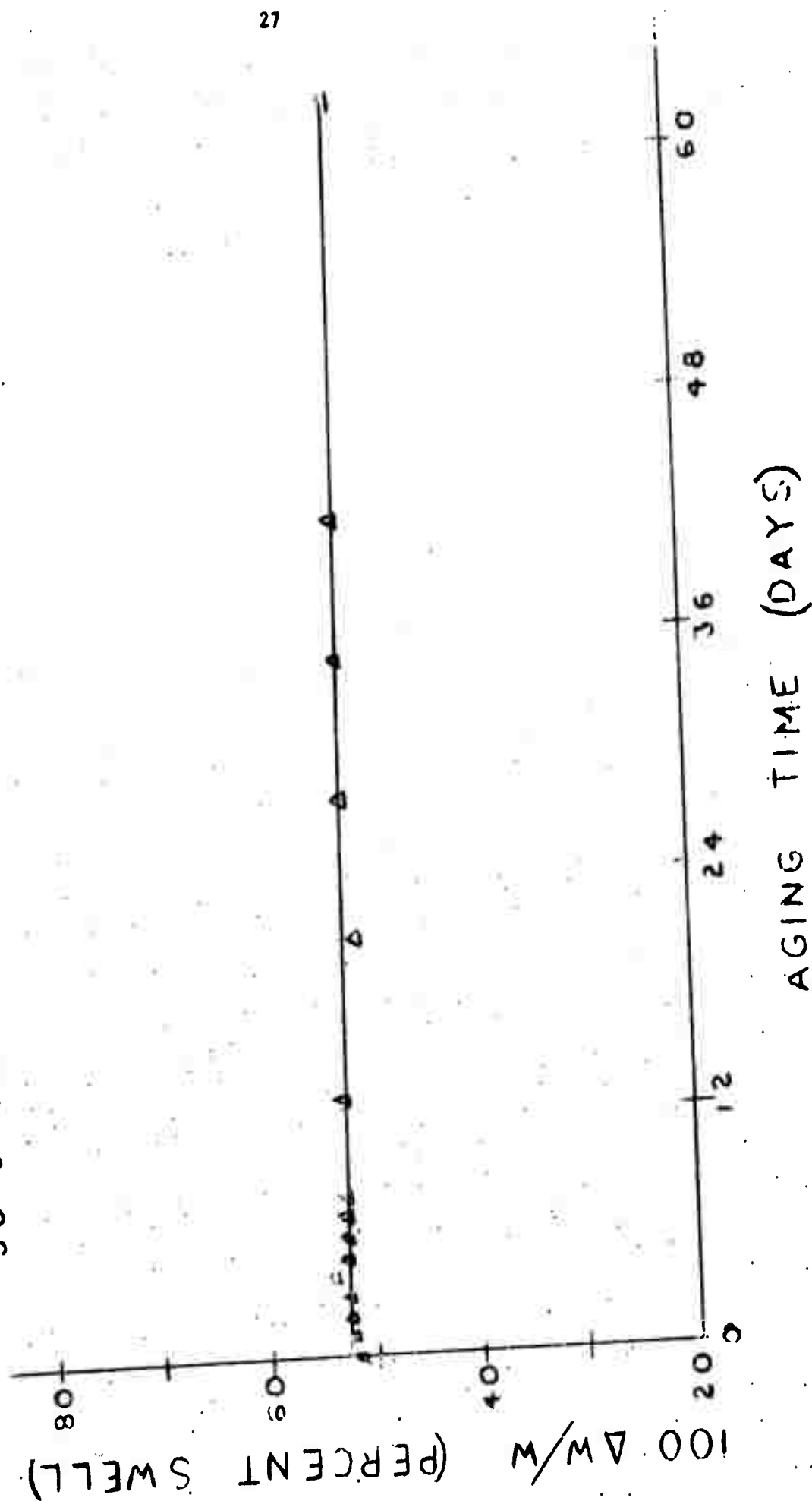


FIGURE 14

SR-822-60 SAMPLES
50°C AGING



of the linear portion of the SR-822-60 curve in Figure 12 with the curve in Figure 13 shows that both curves have the same slope. The only difference between the curves in Figures 13 and 14 is the more gradual slope in Figure 14. This is to be expected since the rubber should age at a slower rate when the aging temperature is decreased.

If one neglects the first portion of the curves in Figure 12 and considers only the linear portion, the curves in Figures 12, 13, and 14 can all be expressed by the mathematical formula for a straight line,

$$y = mx_D + b \quad (1)$$

where m is the slope, b is the y-intercept, x_D is the aging time in days and y is the percentage weight increase due to swelling.

Since the slope of the curve depends on the aging temperature the slope, m , becomes the aging factor.

Solving equation (1) for m gives:

$$m = \frac{y - b}{x_D} \quad (2)$$

which can also be written,

$$m = \frac{[(\Delta W/W) - (\Delta W/W)_0] 100}{t_D} \quad (3)$$

where $(\Delta W/W)_0$ is the percentage weight increase at zero aging time and t_D is the aging time in days. Because $(\Delta W/W)_0$ is usually larger than $(\Delta W/W)$ the curve will generally have a negative slope.

For a given rubber sample the slope, m , is dependent on the aging temperature and the aging stability of the rubber which should be a constant for each type of rubber. Therefore, it can be assumed that m

consists of two factors; the aging temperature factor \bar{T} , and a constant, a , which we shall call the stability constant of the rubber. The temperature factor, \bar{T} , is used instead of the temperature because the temperature functional relationship is not known.

Equation (3) can then be written,

$$a\bar{T} = \frac{[(\Delta W/W) - (\Delta W/W)_0] 100}{t_D} \quad (4)$$

Once a and \bar{T} have been determined for one type of rubber the swelling ability of this rubber at any aging temperature and time can be determined simply by determining $(\Delta W/W)_0$.

It is possible that the rubber may become so brittle if the aging is continued for a sufficiently long enough time that it does not swell $[(\Delta W/W) \text{ goes to zero}]$. However, there is no evidence to indicate that this actually happens. At any rate the percent swelling will decrease until a point is reached where continued aging will not affect the swelling properties of the rubber. At this point the slope goes to zero.

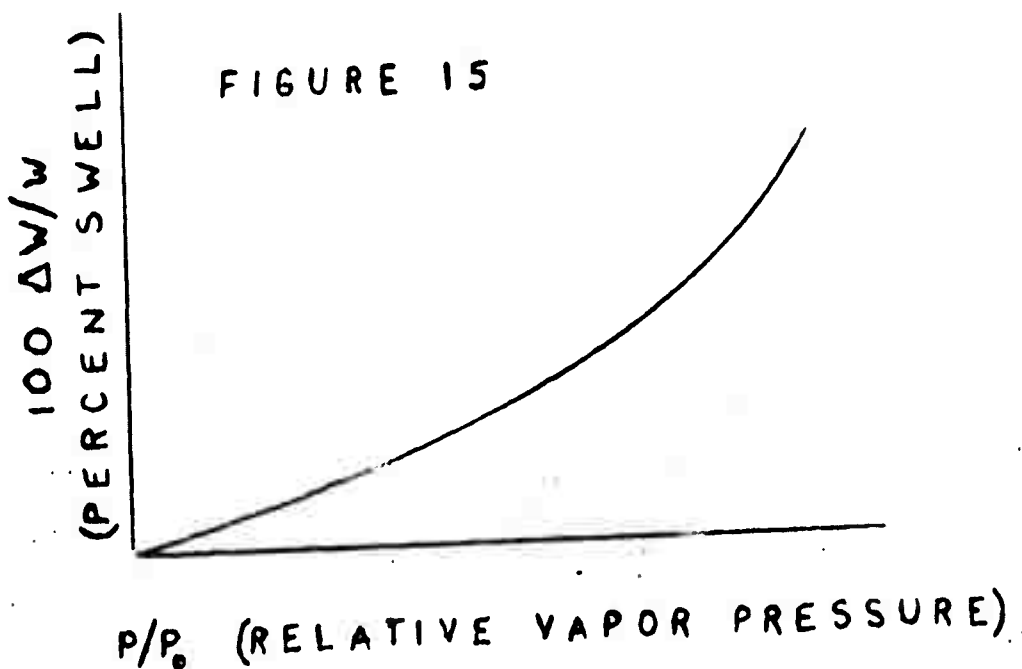
SECTION III

Vapor Phase Swelling Studies with the Quartz Beam Microbalance Apparatus

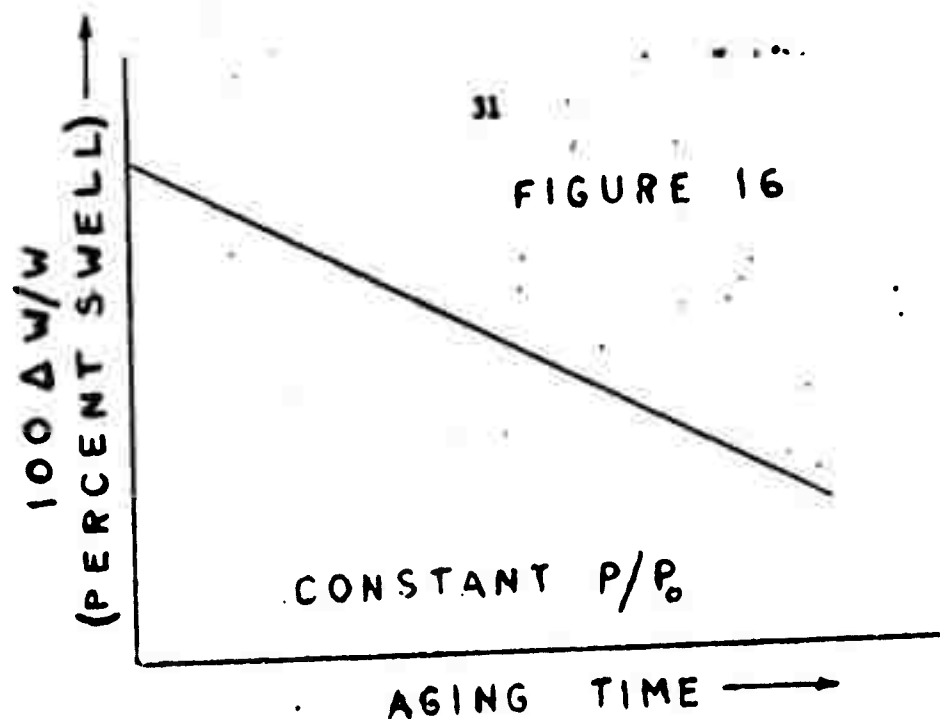
Introduction

The apparatus used in these studies has been described in detail elsewhere. (8) Essentially, the percent swell ($100\Delta W/W$) of the rubber sample is measured as a function of the relative vapor pressure (P/P_0) of the swelling solvent.

The resulting data can be plotted by different methods. The simplest method is shown in Figure 15.



Here, the percent swell of the rubber sample is plotted against the relative vapor pressure of the swelling solvent. Although this graphical method is sensitive to the change in the swelling property with aging, a straight line can be produced by plotting the percent swell of an aged sample (at constant P/P_0) against the time of aging. This relationship is shown in Figure 16.



The equation of the line is given by

$$100\Delta W/W = kt + c \quad (5)$$

where k is the rate constant for the aging process at constant temperature, t is the duration of aging and c is the percent swell of a non-aged sample at constant P/P_0 . The rate constant k is related to the temperature at which the sample was aged by:

$$\log k = \frac{\Delta H}{2.303 RT} + a \quad (6)$$

where; T is the absolute temperature, ΔH is the heat of activation for the aging process, R is the gas constant, and a is a constant of integration. It is evident that a linear relationship exists between $\log k$ and $1/T$. Thus k can be evaluated at the temperature of storage of the rubber sample if two or more values of k are known at higher temperatures. Then the loss in the swelling property at the temperature of storage can be calculated at any time t from equation (5). By combining this data

with the working properties of the rubber, the storage life can be evaluated.

A Comparison of Data for Aged and Non-aged Rubber

The swelling isotherms of rubber O-rings and sheet rubber are shown in Figures 17, 18, and 19 for both aged and non-aged samples. CCl_4 was used as the swelling solvent. It is evident that a decrease in the swelling property occurs under accelerated aging conditions. Complete swelling isotherms were not run on aged rubber O-rings (Figure 19). Instead, the percent swell after different times of aging was measured at only one relative pressure ($0.600P/P_0$). Two of these points are shown in Figure 19.

By plotting the data as in Figure 16 the rate constants for the aging processes at 70°C can be calculated by equation (5). The graphs are shown in Figures 20, 21, and 22. It should be noted that only two points are available in Figures 20 and 21 (from Figures 17 and 18, respectively). In Figure 22, five points are available, although only three of these are indicated in Figure 19. In all cases the points have been taken at a relative vapor pressure of 0.600. The rate constants are presented in Table VI.

TABLE VI

<u>Rubber Type</u>	<u>Rate Constant at 70°C</u>
HN-81848-R sheet	- 0.031 day ⁻¹
HN-80496-DSP2 sheet	- 0.076
SR-822-60 O-rings	- 0.033

The data indicate that HN-81848-R sheet rubber and SR-822-60 rubber O-rings age at about the same rate while HN-80496-DSP2 sheet

FIGURE 17

SWELLING OF BN-81848-R BY CCl_4 AT 20°C

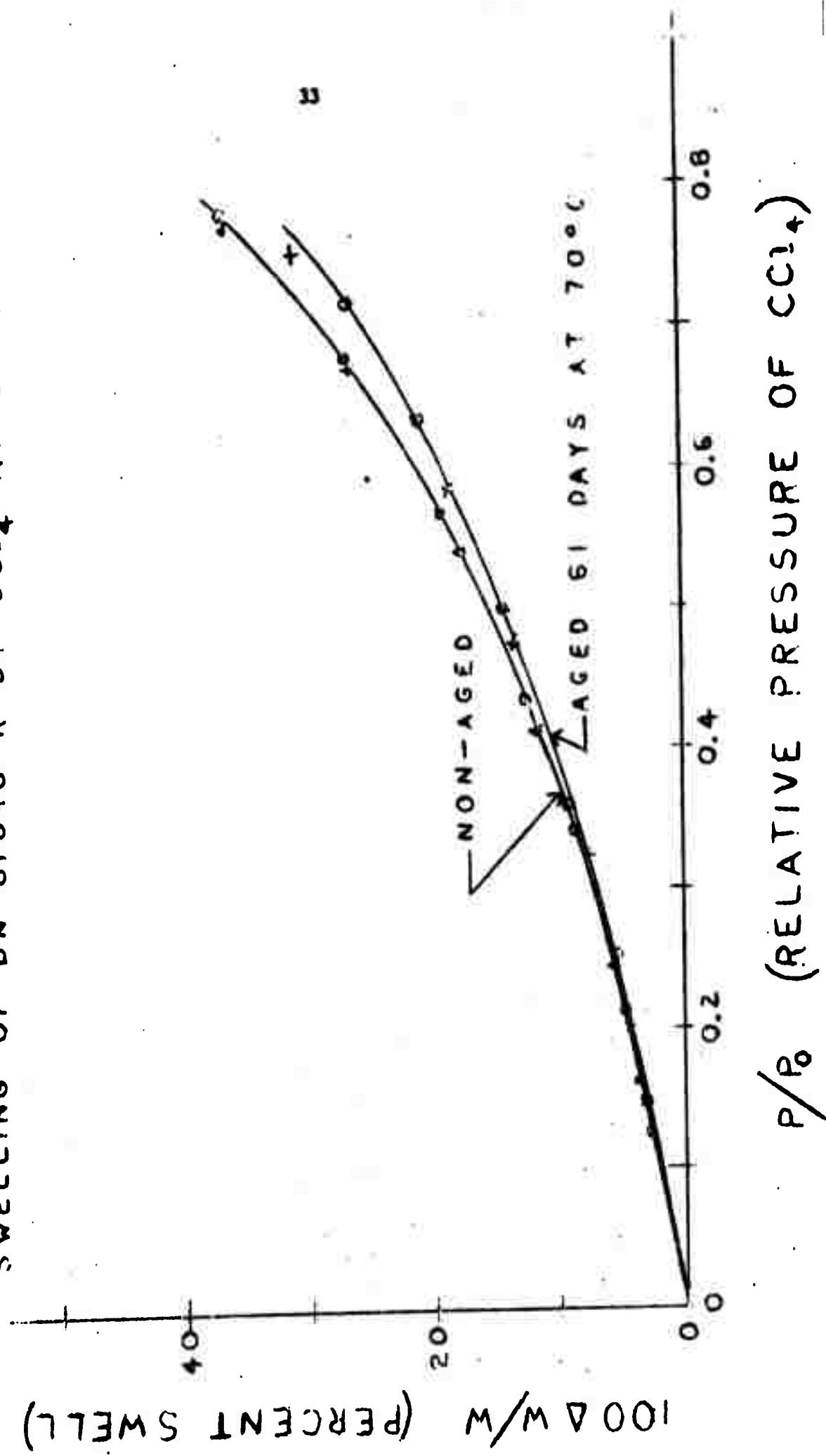


FIGURE 18

SWELLING OF BN-80496-DSP2 BY CCl_4 AT 20°C

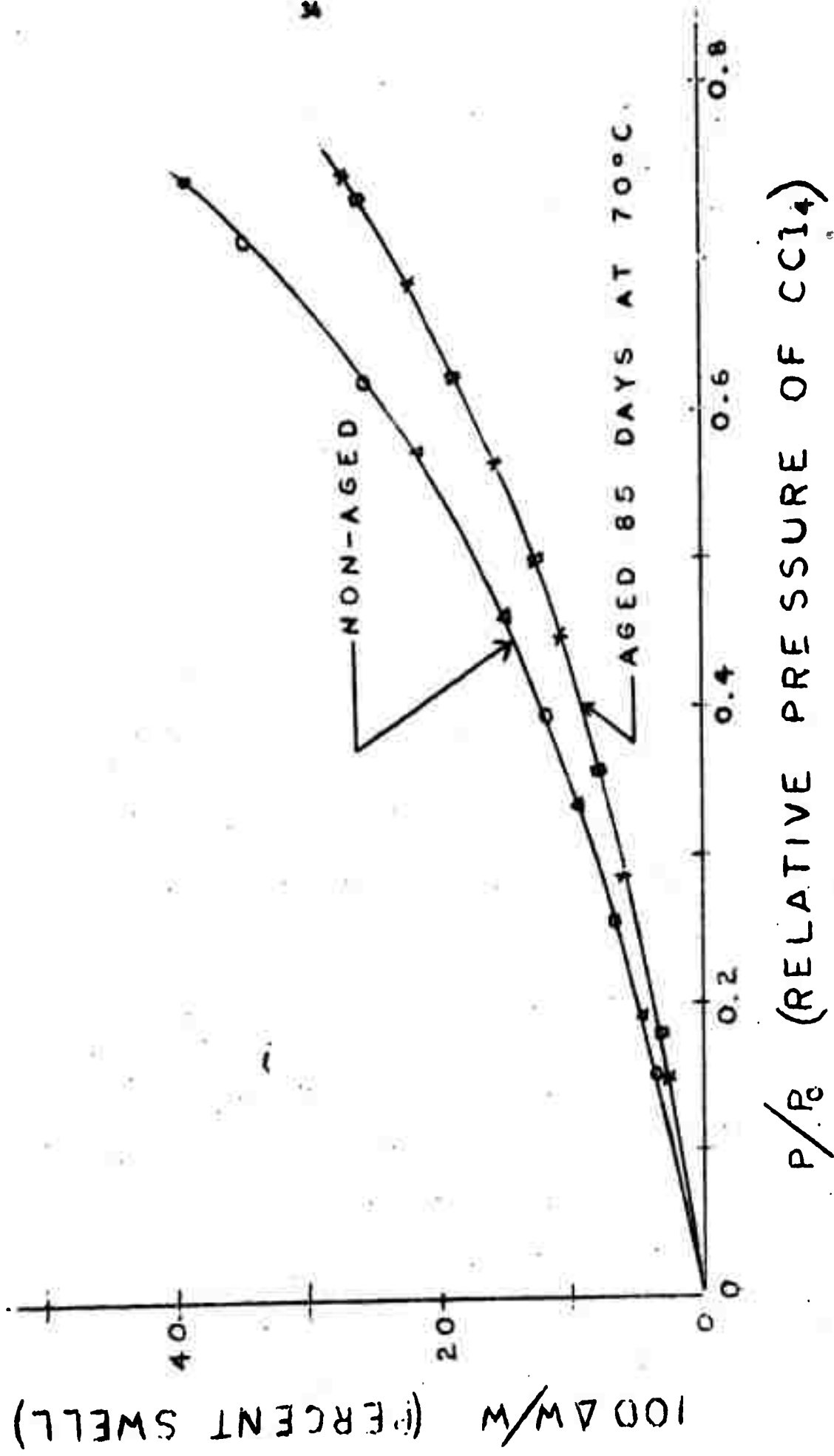


FIGURE 19

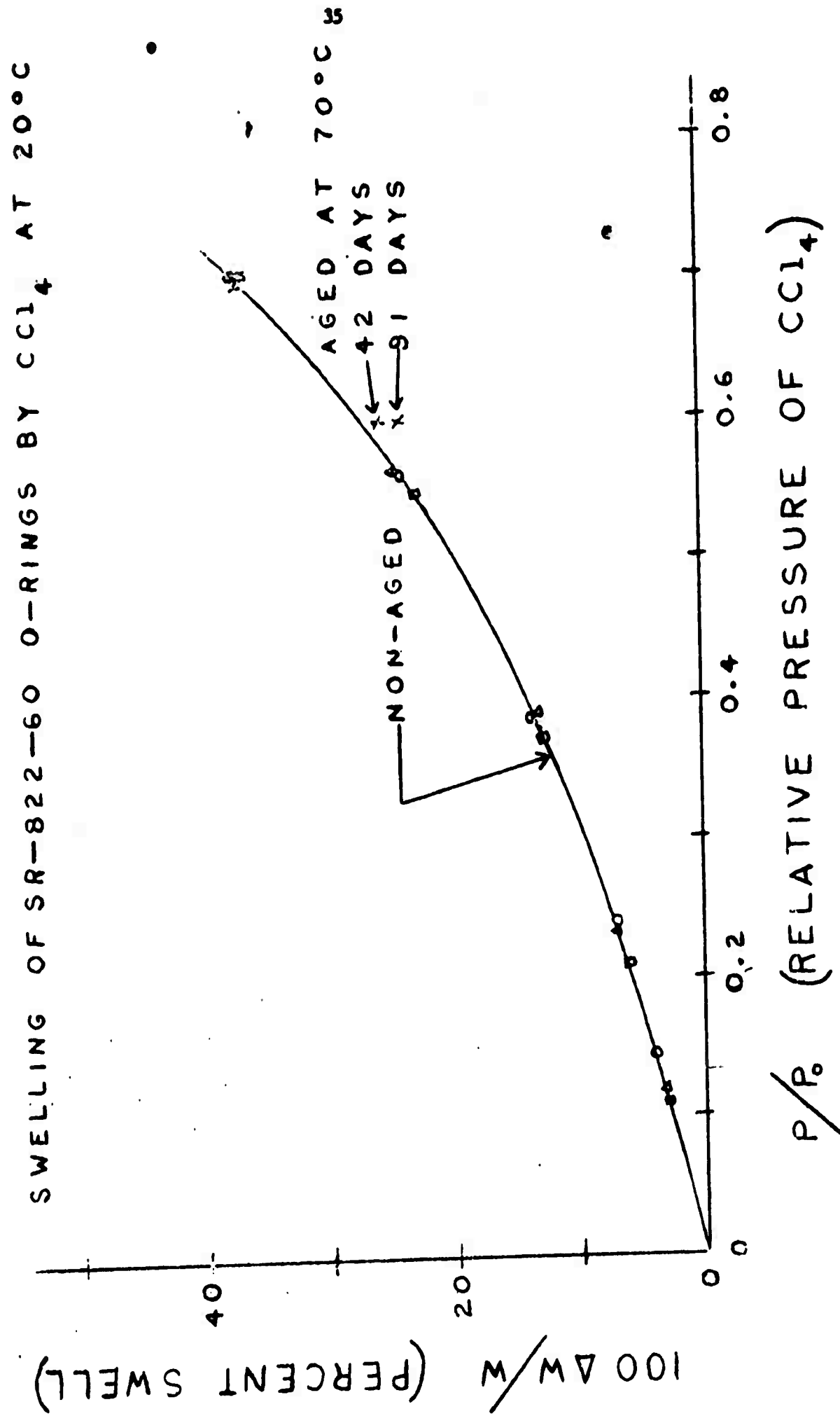


FIGURE 20

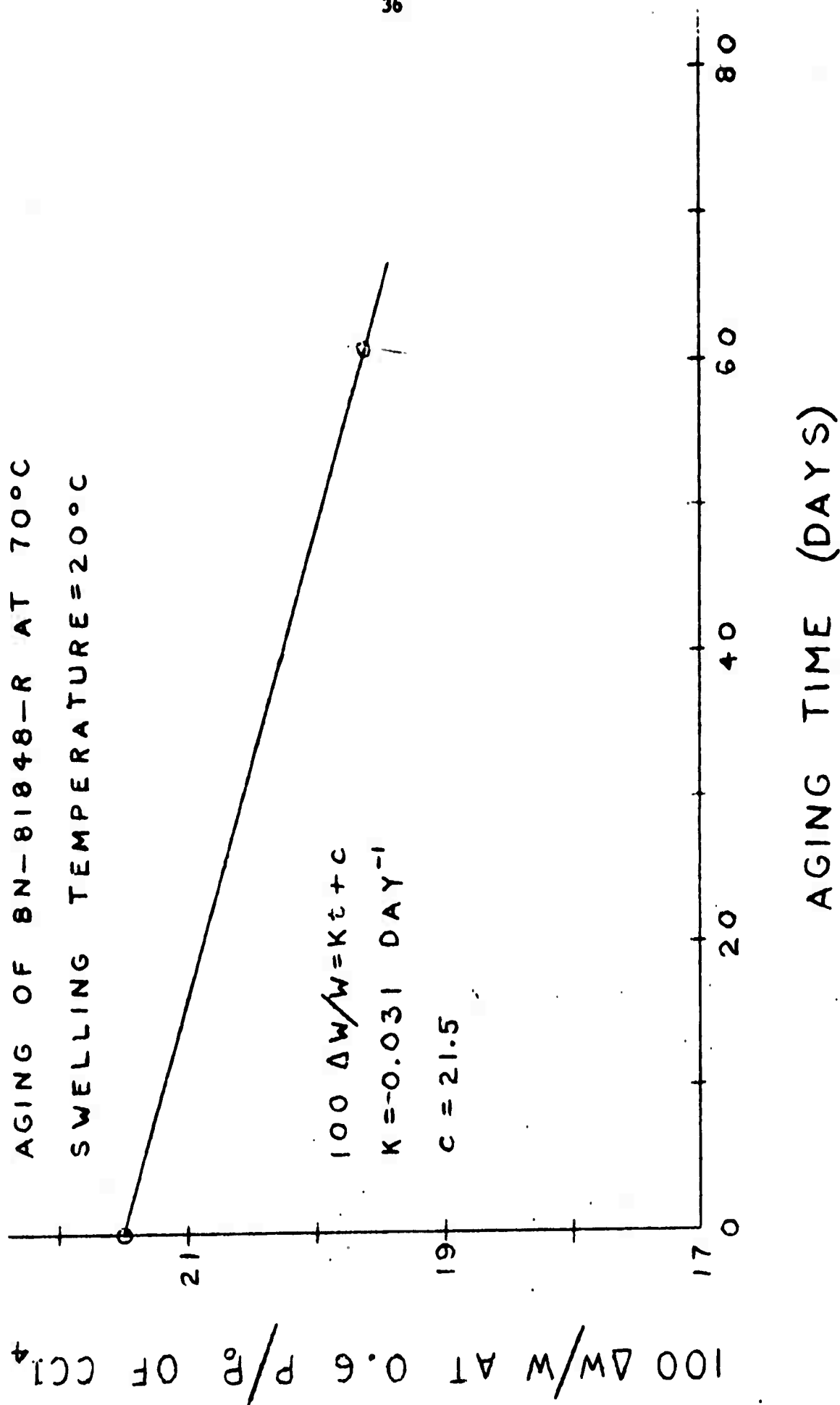


FIGURE 21

AGING OF BN-80496-DSP2 AT 70°C

SWELLING TEMPERATURE=20°C

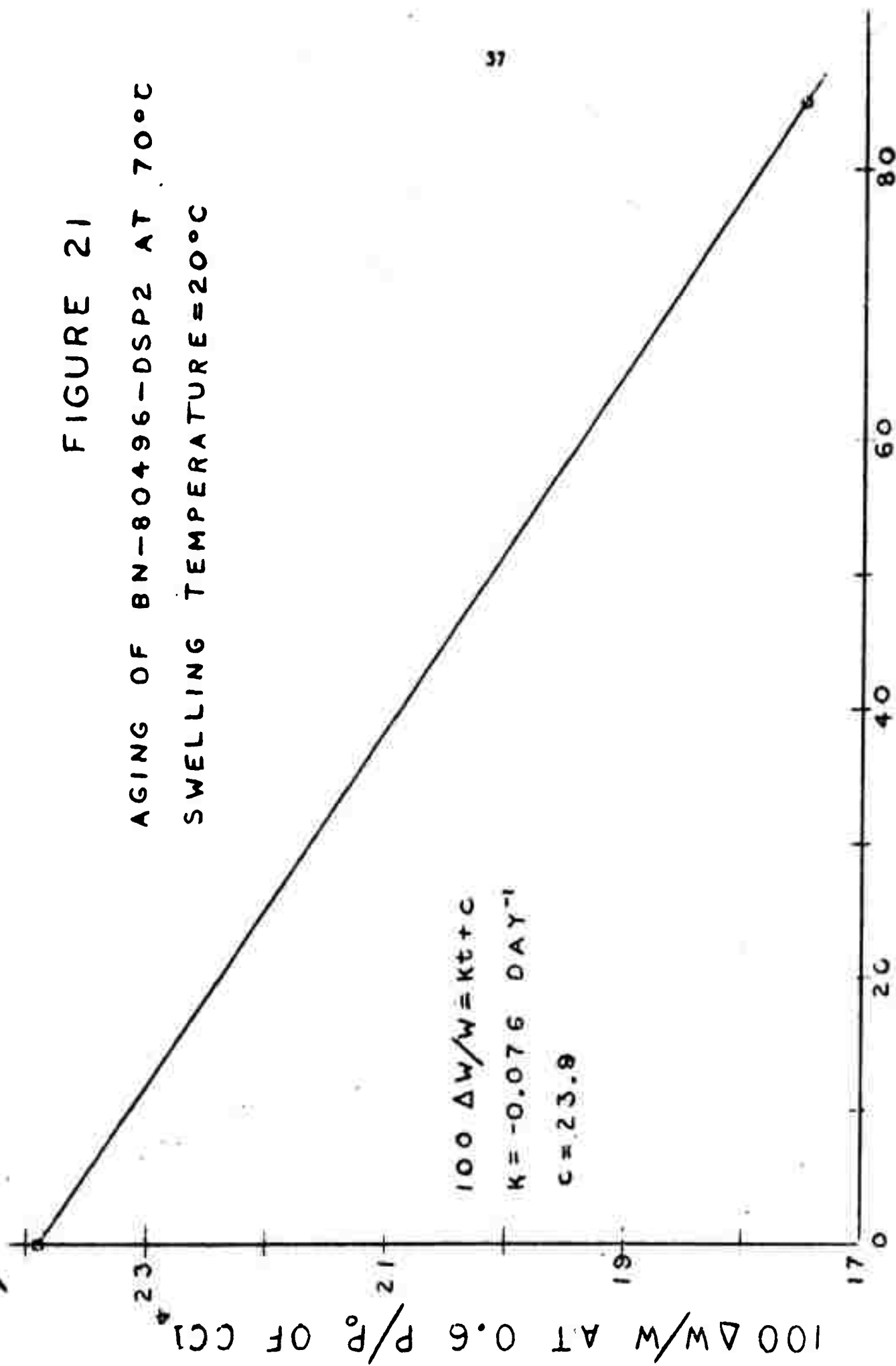
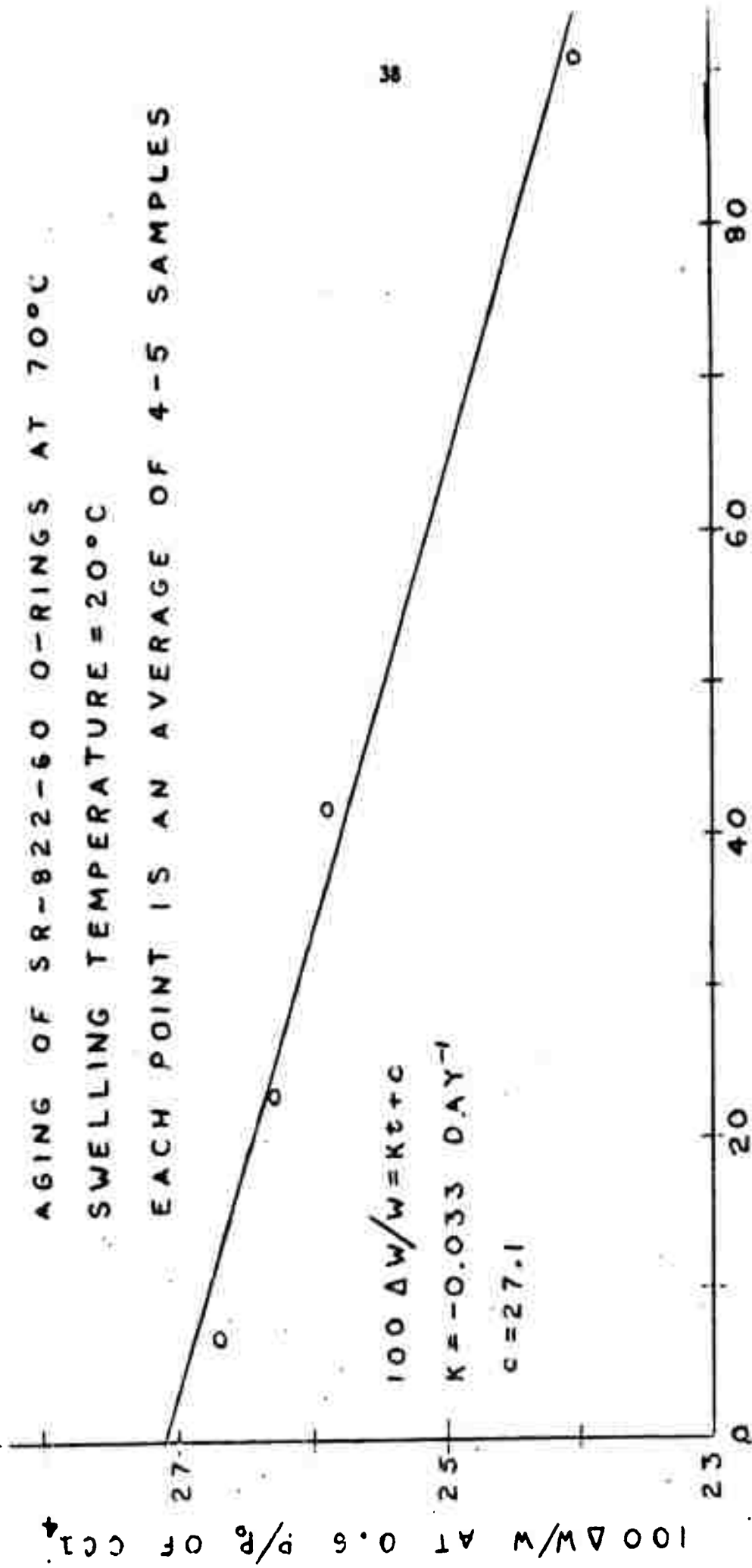


FIGURE 22

AGING OF SR-822-60 O-RINGS AT 70°C

SWELLING TEMPERATURE = 20°C

EACH POINT IS AN AVERAGE OF 4-5 SAMPLES



rubber ages more than twice as fast as the other two.

Because the aging data is available at only one temperature (70°C) the rates of aging at the temperature of storage cannot yet be calculated. Samples of rubber O-rings are presently being aged at 90°C to allow this calculation.

Theoretical Studies on the Aging Process

A theoretical investigation of the aging process should allow the evaluation of factors responsible for the break-down of rubber and permit the selection of rubber compounds better suited for a particular function. For these reasons an attempt is being made to interpret the swelling data according to the theory of Flory ⁽⁹⁾. This theory predicts that swelling data should fit the equation

$$2.303 \log \frac{P/P_0}{1-V_2} - V_2 = X_1 V_2^2 \quad (7)$$

where P/P_0 is the relative vapor pressure of the swelling solvent, V_2 is the volume fraction of the rubber in the rubber-solvent gel, and X_1 is a constant called the mixing parameter. By plotting

$2.303 \log \frac{P/P_0}{1-V_2} - V_2$ against V_2^2 a straight line should result, with a slope equal to X_1 . Plots have been made for the swelling data on both aged and non-aged sheet rubber (BN-80496-DSP2). The results are shown in Figures 23 and 24. Although some data scatter is present, the points fall reasonably well on a straight line. For non-aged samples X_1 equals 0.695. For samples aged 85 days at 70°C, $X_1 = 0.895$. Thus, the value of X_1 is sensitive to the break-down of the rubber samples.

FIGURE 23

SWELLING OF NON-AGED
8N-80496-DSP2 BY CCl₄

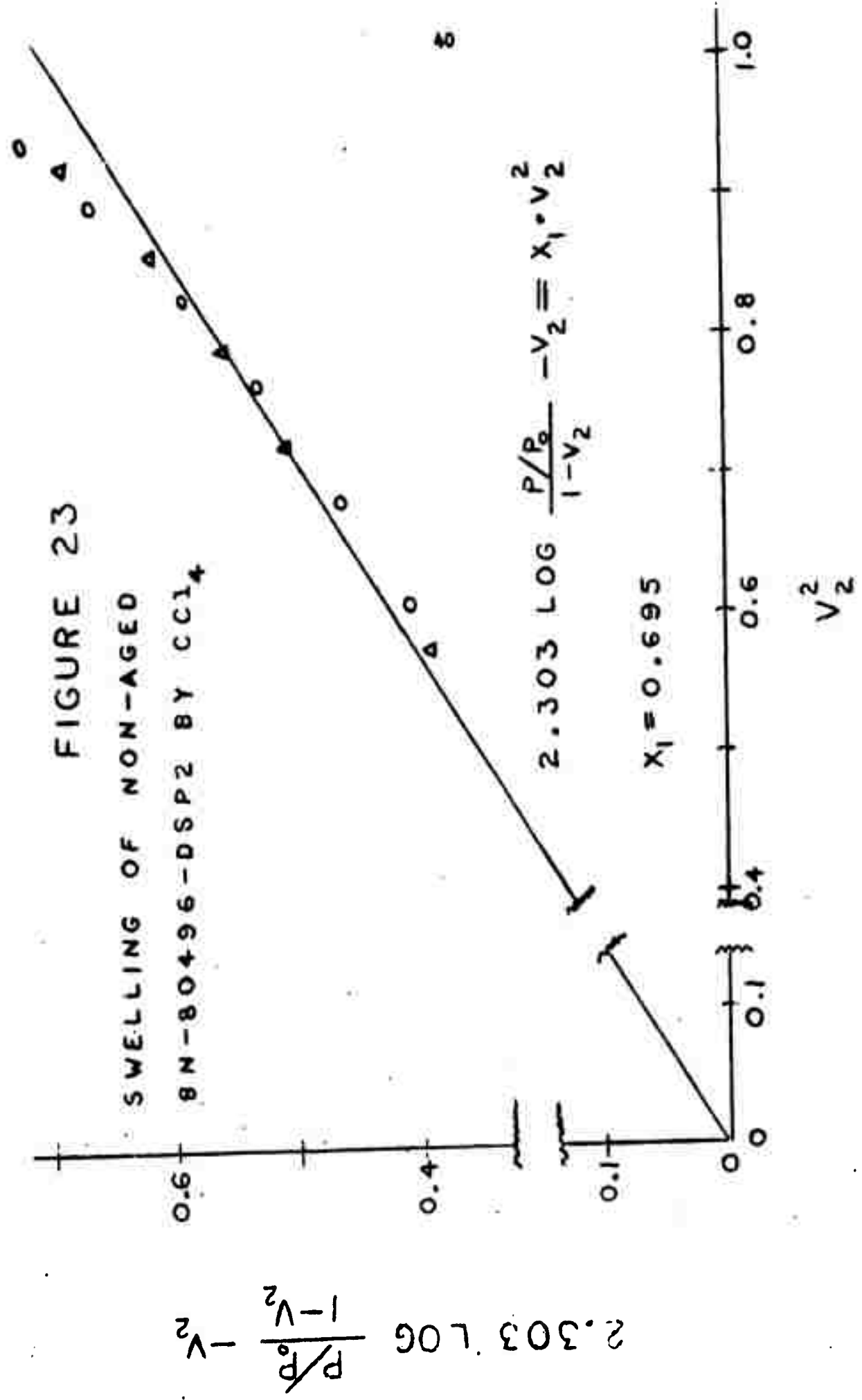
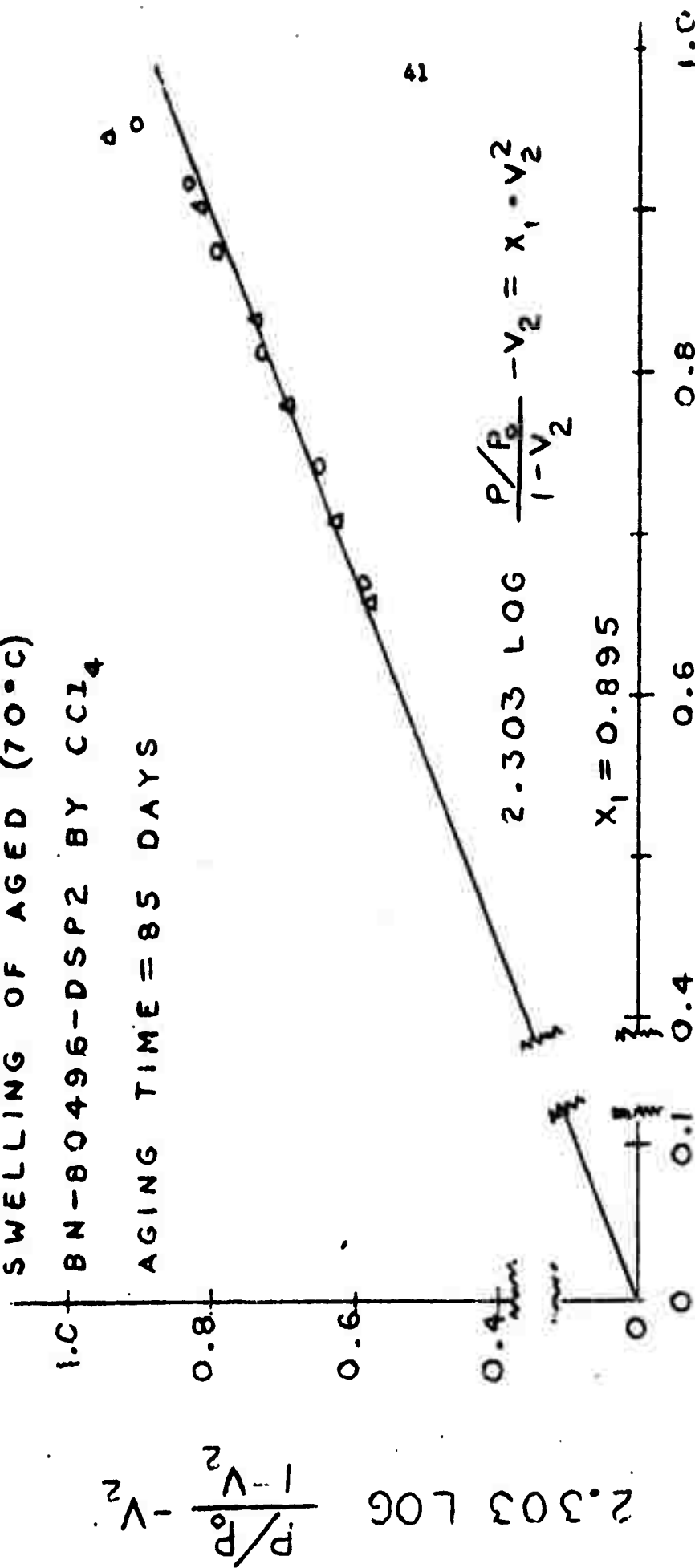


FIGURE 24

SWELLING OF AGED (70°C)

BN-80496-DSP2 BY CCl_4

AGING TIME = 85 DAYS



A second means of plotting the data is shown in Figure 25. In this graph the points represent the experimental data and the curves are calculated from the values of X_1 . It is evident that the results are different for aged and non-aged samples.

According to the theory, X_1 is related to the heat of dilution, $\Delta \bar{H}_1$, of the solvent in the rubber by

$$\frac{\Delta \bar{H}_1}{RT} = X_1 V_2^2 \quad (8)$$

where $\Delta \bar{H}_1$ is given by the relationship

$$\log P/P_0 = - \frac{\Delta \bar{H}_1}{2.303 RT} + C' \quad (9)$$

Thus, by measuring P/P_0 at constant V_2 as a function of the absolute temperature $\Delta \bar{H}_1$ and X_1 can be calculated. Providing the theory is correct, the value of X_1 as calculated from equation (8) should be the same (within the limits of experimental error) as the value calculated from equation (7). Alternatively, $\Delta \bar{H}_1$ can be calculated (at constant V_2) from equation (8) with the value of X_1 from equation (7). This value of $\Delta \bar{H}_1$ should then correspond to the measured value (from equation 9). Although the experimental technique in measuring $\Delta \bar{H}_1$ has not yet been perfected, the values obtained thus far are definitely of the right magnitude (150-300 cal/mole).

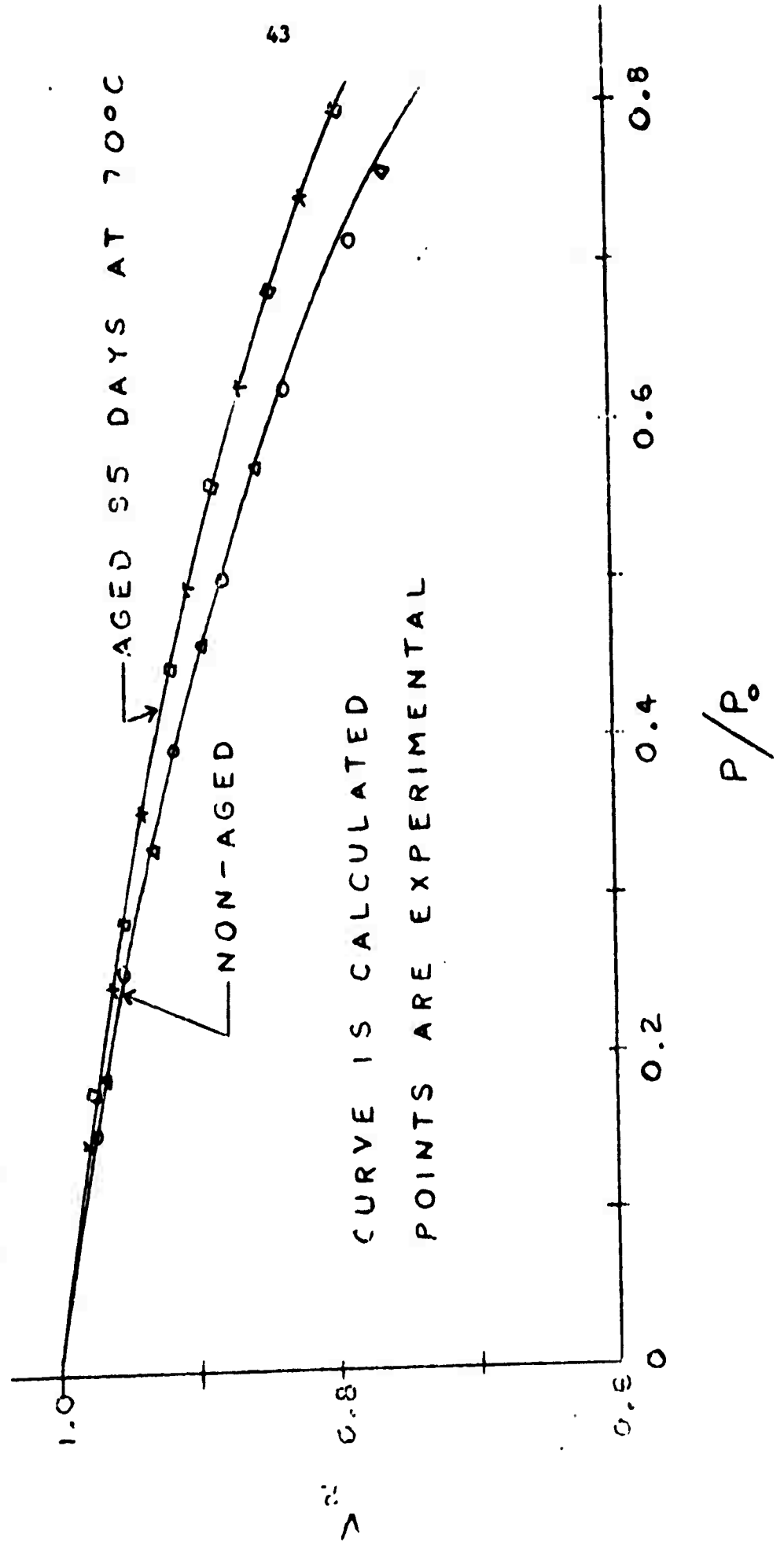
Future Work on Swelling Studies

Heat of dilution measurements will be continued as a further test of the Flory theory. Also, swelling data will be obtained on rubber O-rings aged at different temperatures. With this data the rate of loss in the swelling property at the temperature of storage will be calculated.

FIGURE 25

SWELLING OF BN-80496-DSP2 BY CO_2

SWELLING TEMPERATURE = 20°C



By combining the swelling data with data on other physico-mechanical properties (modulus, relative elongation, etc.) it should be possible to calculate the storage life of the O-rings.

SECTION IV

**Vaporization of Volatile Components
In the Rubber**

A rubber sample measuring approximately 1 cm X 2 cm X 2mm and weighing approximately 0.6 gram was suspended in a drying oven maintained at a constant temperature of 70°C. The sample was suspended near the bulb of the thermometer to insure that the temperature in the vicinity of the sample remained at the desired temperature. Air was allowed to circulate freely through the oven by means of an opening in the top of the oven. The sample was removed from the oven at different intervals, allowed to cool in air for ten minutes, weighed with an analytical balance and returned to the oven. The sample used in this study was cut from the same sheet of SR-822-60 rubber that was used to obtain the curves in Figures 13 and 14. Results of this experiment are given in Figure 26, which is a plot of percentage weight decrease of the sample, $-(\Delta W/W)100$, versus aging time in hours.

Two curves are shown in Figure 26. Curve A is a plot of the data obtained in the experiment. This curve shows that there is a rapid decrease in weight during the first few hours of aging, probably due to vaporization of moisture and grease picked up by the sample during storage and handling. Beyond this point the curve becomes linear. If one assumes that the first portion of this curve is due to vaporization of grease and moisture (to be verified in future experiments) and that the weight loss with aging time is a linear function as indicated by the remainder of the curve, then the plot can be corrected for the presence of these foreign materials. This is done by extrapolating the linear portion of the curve to zero time, taking another point on curve A and subtracting the value determined by extrapolating the curve to zero time. A straight line can then be constructed through the two points,

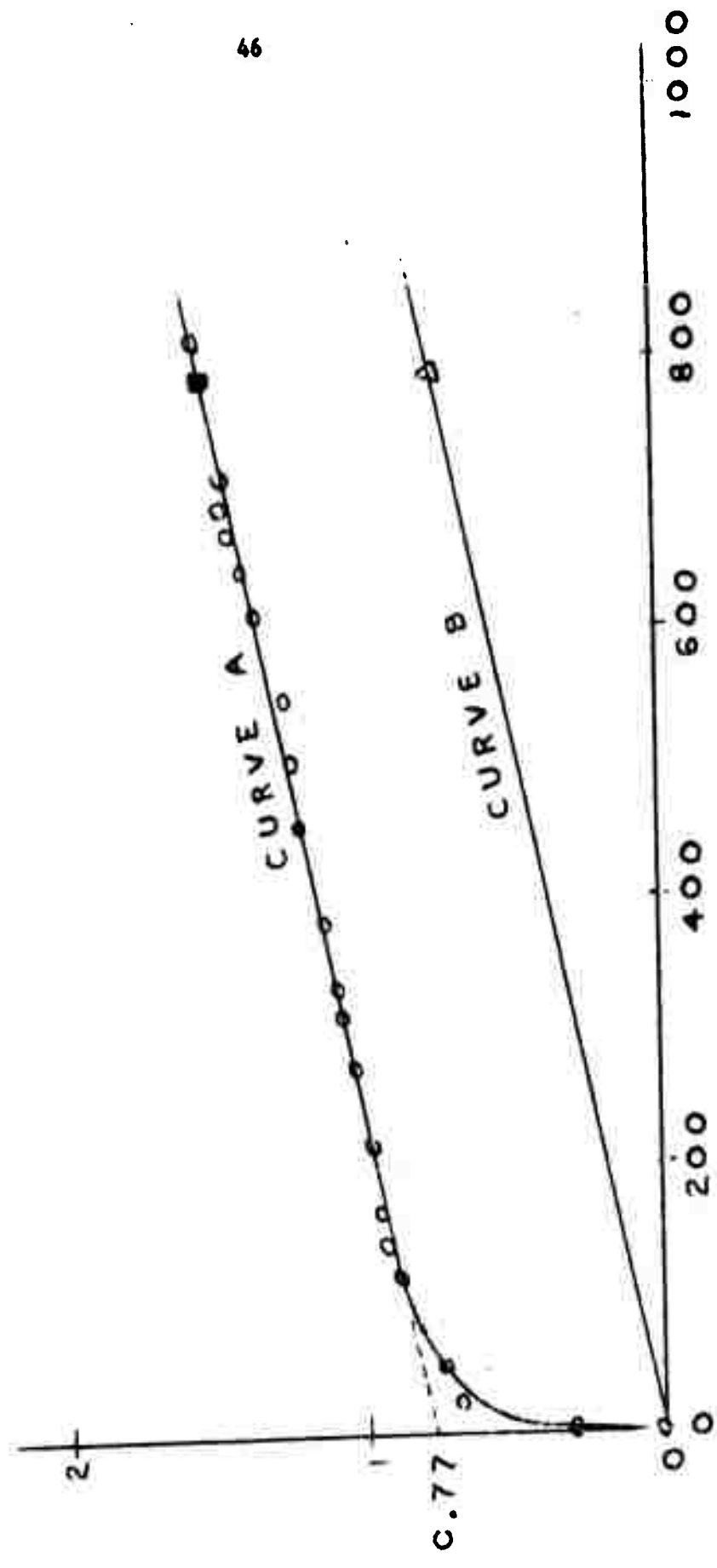
FIGURE 26

SR-822-60 SAMPLES AGED AT 70°C

O = DATA CURVE

Δ = TRUE AGING CURVE

-100 ΔW/W (PERCENT WEIGHT LOSS)



one of which becomes the origin. Curve B in Figure 26 is the corrected curve and should be the true volatilization curve. This procedure in no way alters the slope of the line.

SECTION V

Neutron Irradiation of Rubber

In an attempt to learn more about the dependence of swelling on crosslinking several samples were placed in the University of Oklahoma research reactor and bombarded with neutrons.

Two samples of a Buna-N type rubber of the same size and composition as those used in the swelling studies were placed in the reactor at a flux position of 10^8 neutrons/cm²/second. The samples were bombarded for 240.45 watt-hours, removed from the reactor (radiation level of samples was negligible) and swelled in the modified test tube apparatus for 72 hours using benzene as the solvent at a temperature of 20°C.

In addition to these two samples three control samples that had not been bombarded were swelled under the same conditions. Experimental results are given in Table VII.

Sample 1 was first swelled for 72 hours, allowed to deswell and then was placed in the reactor in an attempt to see if swelling changed the crosslinking or if the presence of solvent molecules during bombarding affected the degree of crosslinking. After being bombarded the sample was again swelled along with the other four samples.

TABLE VII

Bombarded Samples

<u>Sample Number</u>	<u>Weight Before Bombarding (grams)</u>	<u>Weight After Bombarding (grams)</u>	<u>Weight Of Swelled Sample (grams)</u>	<u>Weight Increase (grams)</u>	<u>Percent Weight Increase</u>
1	0.9915	0.9829	1.6925	0.7096	72.19
2	0.9564	0.9564	1.6542	0.6978	72.96

Samples Not Bombaraded

Sample Number	Original Sample Weight (grams)	Weight Of Swelled Sample (grams)	Weight Increase (grams)	Percent Weight Increase (grams)
3	0.6885	1.2152	0.5267	76.50
4	0.6109	1.0662	0.4553	74.53
5	0.7503	1.3251	0.5748	76.61

As a control to see if swelling, deswelling and reswelling changed the percent weight increase, sample 5 was subjected to the same conditions as sample 1 but was not bombarded.

Examination of Table VII shows that there appears to be a decided change in the swelling ability of the rubber sample after neutron bombardment. If the bombardment were continued for a sufficiently long period of time enough crosslinking would occur to render the sample brittle and useless. The results obtained when sample 4 was swelled detract from the total results since this sample would make it appear that the percentage swell of all the samples is approximately $74 \pm 2\%$. Further experimentation would be necessary to show that the results from this sample can be disregarded.

Due to the lack of time, money and facilities this study was not pursued past the initial stages. There are many complicating factors that arise in a problem such as this and fruitful results can be obtained only with the proper equipment and an accurate knowledge of the material subjected to irradiation. Since none of these were available when the problem was started it was decided that preliminary studies would be made and then set aside to concentrate on more fundamental work with the intention of returning to this program at a future date if possible.

SECTION VI

Swelling Behavior Under Stress Conditions

In another experiment designed to study the degree of crosslinking of an elastomer as well as its swelling behavior under stress, a stretching rack was designed that would permit rubber strips to be stretched to any desired elongation. The rack used in this experiment is shown in Figure 27.

The frame, f, is made of aluminum of single piece construction measuring 55 mm X 15 mm X 5 mm. A turnscREW, s, passes through the upper portion, b, of the frame, which also acts as a holding base, and is secured to a movable base, b'. The rubber sample is secured to the frame between base, b, and the removable clamp, r, in the upper portion of the frame and between base b' and removable clamp r' in the lower portion of the frame. Two screws pass through each removal clamp and are screwed into bases b and b' to hold the sample in place. The sample is stretched to the desired elongation by turning screw s, which causes the movable base, b', to move down the frame. The sample can then be swelled by suspending the frame from hook t in the modified test tube apparatus (Figure 9).

When making a determination rubber samples measuring approximately 20 mm X 5 mm X 2 mm were secured to the frame and swelled at zero elongation. The samples were then allowed to deswell, stretched to 100 percent elongation and reswelled. The strain placed on the rubber sample due to stretching and swelling has caused every sample that has been used to date to break. The same results were obtained when a sample was stretched and aged at elevated temperatures. Stretching the sample to an elongation small enough to prevent breakage causes such a small change in the swelling ability of the sample that one can conclude that no change is found within experimental error.

Since aircraft engines and other Air Force equipment using rubber O-rings are stored in 1010 oil until needed it was considered necessary to determine what effect the 1010 oil had on O-rings that were saturated with this oil for long periods of time. In order to do this it was first necessary to find a method for removing the 1010 oil without causing a change in the properties of the rubber due to the method itself.

Placing O-rings saturated with 1010 oil in a reduced atmosphere system had very little effect on the oil because of its low vapor pressure. Heating the system to a temperature of 50°C for five days failed to remove more than a few milligrams of oil from the rings. Since heating the rubber samples changes the properties of the rubber and because such long periods of time are required to remove all the oil in a simple vacuum system this method was considered impractical. Attempts to remove the oil by soaking the rings in organic solvents or exposing the rings to solvent vapors and then evaporating the solvent from the rings under vacuum likewise failed because most organic solvents remove soluble constituents from the rubber along with the 1010 oil. Even soaking the rings in water containing a detergent caused solution of constituents from the rubber.

The method that appears most promising consists of placing the oil soaked rubber sample in a high vacuum system made up of an evacuating chamber, cold trap, mercury diffusion pump and fore pump with a minimum of connections. The rubber samples are placed in the evacuating chamber, held under vacuum for three days and weighed. Experimental results are given in Figure 28, where the sample weight is plotted versus the total number of days the sample was subjected to high vacuum.

SECTION VII

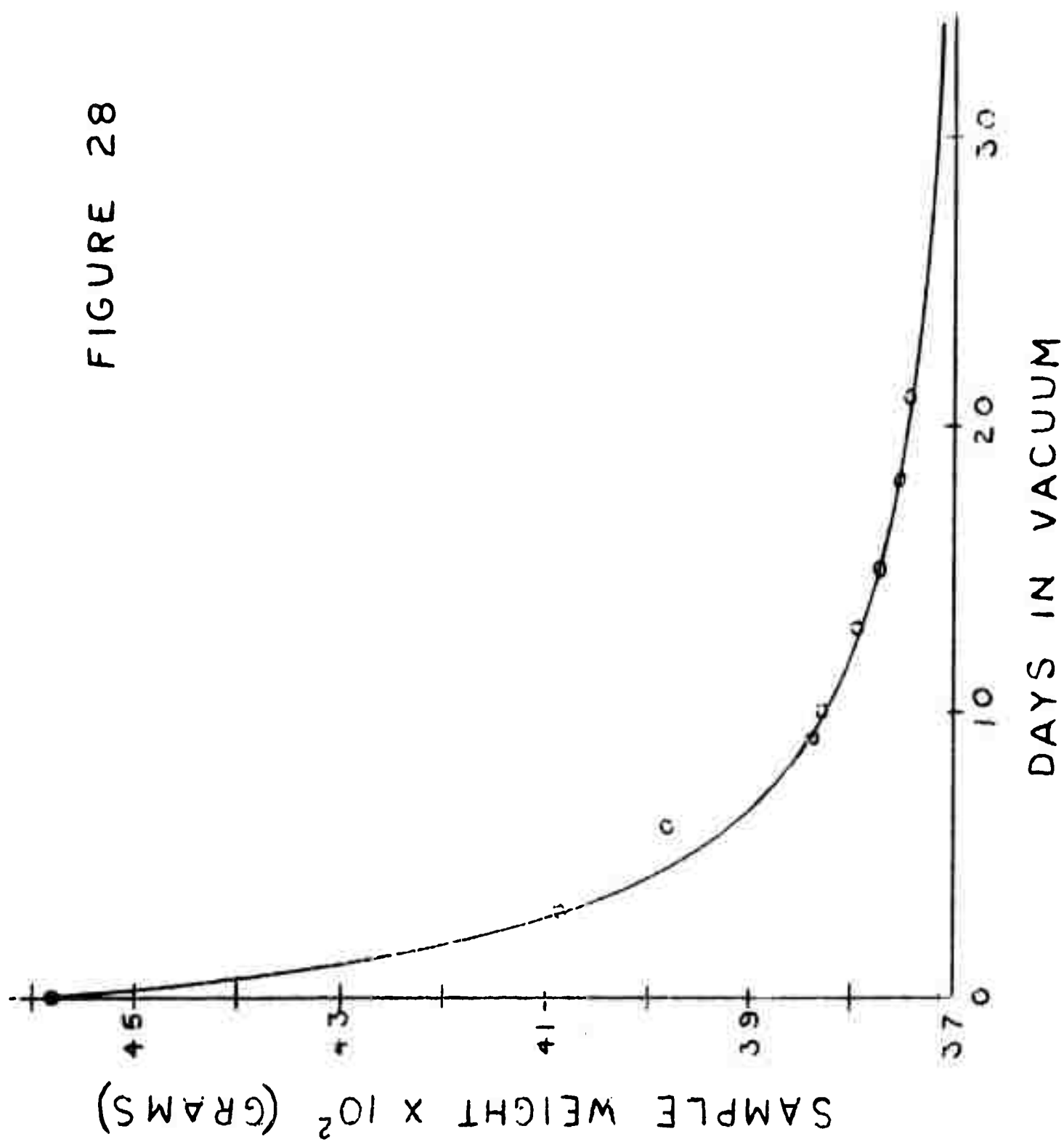
Removal of 1010 Oil from O-Rings

Since aircraft engines and other Air Force equipment using rubber O-rings are stored in 1010 oil until needed it was considered necessary to determine what effect the 1010 oil had on O-rings that were saturated with this oil for long periods of time. In order to do this it was first necessary to find a method for removing the 1010 oil without causing a change in the properties of the rubber due to the method itself.

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FIGURE 28



According to this graph the sample loses weight rapidly during the first ten days in the system and then gradually approaches a constant weight, achieving this constant weight in about five weeks.

To insure that the vacuum did not remove volatile components from the rubber and thereby change its properties, a control sample that had not been soaked in oil was placed in the vacuum system. At the end of eight days in the system the sample weight had been reduced by approximately 0.8 percent. This weight loss most likely represents the removal of moisture and grease present in the rubber due to handling and storage.

Once this method has been perfected the physical properties of O-rings that have been saturated with 1010 oil and then had the oil removed will be compared with the physical properties of control samples of the same type of rubber that have not been soaked in oil to see what effect the 1010 oil has on the properties of the rubber.

SECTION VIII

Leaching of Constituents from O-Rings

In an attempt to learn more about the effect of accelerated aging on rubber samples a technique was devised to study the material leached from a rubber sample immersed in carbon tetrachloride by means of infrared spectrum analysis. The basis for the experiment was the possibility that aging affected the soluble materials in a definite manner and this change would be evident by changes in the infrared spectrum.

Rubber samples of two different types of Buna-N rubber were heated in air at 70°C for periods ranging from $1\frac{3}{4}$ hours to eight days. The rubber samples measured about 1 cm X 1 cm X 3 mm and weighed approximately 0.4 gram. When a sample was removed from the oven it was allowed to cool overnight, immersed in 50 ml of carbon tetrachloride for 72 hours and removed.

Since the amount of solvent present greatly exceeded the amount of leached material, solvent bands would mask bands due to the soluble material. To eliminate this, and to have a more concentrated solution, the solvent was evaporated by passing a stream of air over the solution contained in a beaker. As soon as the solvent had been completely evaporated the walls of the beaker were washed down with a small amount of solvent and the solution was carefully transferred to a sodium chloride disc where the solvent was again evaporated using compressed air. This was done three times to insure complete transfer of material to the disc. A second sodium chloride disc was placed over the first so the material was contained between the discs. The discs were then placed in a clamp which held the discs in the infrared beam.

Because all the samples used could not be subjected to identical conditions in making these determinations it was necessary to find an internal standard that could be used as a comparison against all other peaks in the spectrum. In other words it was necessary to find a peak that remained constant in relation to the other peaks in the same spectrum.

Extensive tests have been made using this technique but to date no definite correlation has been found between the infrared spectrum of the leached materials and the aging time. It is suspected that the reason for this is the difficulty involved in trying to duplicate the same conditions from sample to sample. The weights vary from sample to sample and may introduce some error although use of an internal standard should eliminate this. The most probable cause for error arises in transferring the solution from the beaker to the disc. In future studies a technique will be devised to eliminate this step from the procedure.

SECTION IX

Temperature Retraction Apparatus

The Temperature Retraction test (ASTM D 1329-60) provides a method for rapid evaluation of crystallization effects and for comparing viscoelastic properties of rubber and rubber-like materials at low temperatures.

Apparatus

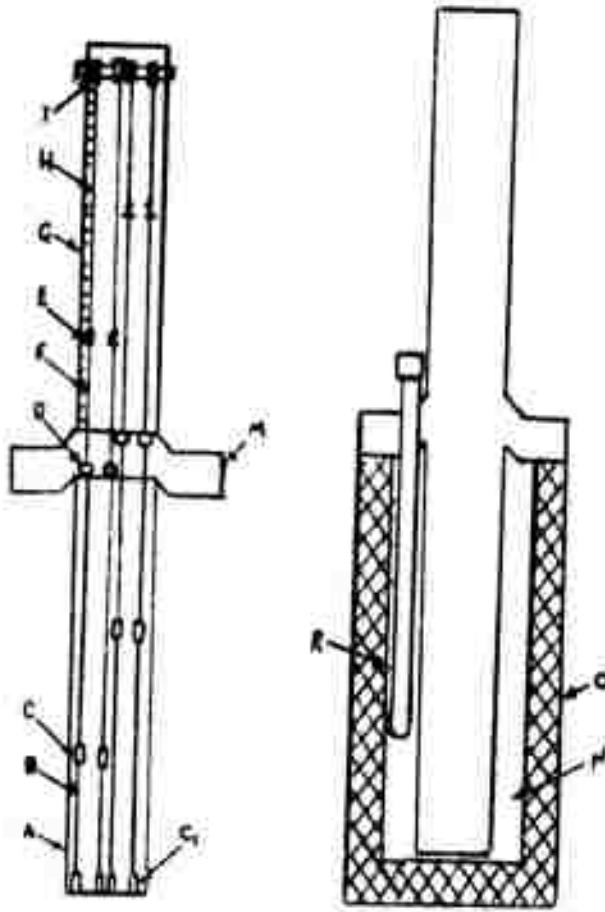
The testing apparatus consists of a specimen rack, an insulated cooling bath which is equipped with a thermometer, an immersion heater, and a liquid coolant. A schematic drawing of the apparatus is shown in Figure 29. The specimen rack was designed so that it maintains a slight tension (1 to 3 psi) on the specimens and permits them to be stretched and anchored at any elongation up to a maximum of 250 percent. The length of the specimens can be read by an indicator at any time during the test within an accuracy of ± 0.05 centimeters. The rack was designed so that it can hold four specimens at the same time.

The bath consists of a silvered Dewar flask which sets in a wooden box. The liquid coolant equilibrium mixture used is methanol and dry ice.

Procedure

The Dewar flask is filled three-fourths full with the methanol-dry ice equilibrium mixture at about -70°C by adding chopped dry ice to methanol. One end of the test specimens are inserted into the stationary clamp at the bottom of the sample rack and the other end in the movable clamp. The three inch samples are then stretched to the desired length and anchored in the elongated position by tightening the thumb nuts. The specimen rack is placed in the bath slowly to avoid frothing.

FIGURE 29



A - Specimen Rack
 B - Test Specimen
 C - Movable Clamp
 C₁ - Stationary Clamp
 D - Thumb Nut
 E - Indicator
 F - Connecting Wire

G - Graduated Scale
 H - Flexible Cord
 I - Pulley
 M - Rack Support
 N - Bath
 O - Wooden Container
 R - Heater

If the temperature of the bath rises above -70°C when the rack is inserted, a small amount of dry ice is added to reduce the temperature to between -70° and -73°C . After 10 minutes the thumb nuts are released, and the specimens are allowed to retract freely. At this point the heater is turned on. The first reading is taken at -70°C , and continued at two minute intervals until retraction is complete.

Preliminary Results

Data is given below in Table VIII and in Figure 30 on three Bama-N rubber samples of different cure date (not the same rubber compound in all three samples). Three inch samples are given an initial elongation of five centimeters (65.7% testing elongation) and placed in a methanol-dry ice bath at -70°C for 10 minutes. The samples are then released and allowed to retract.

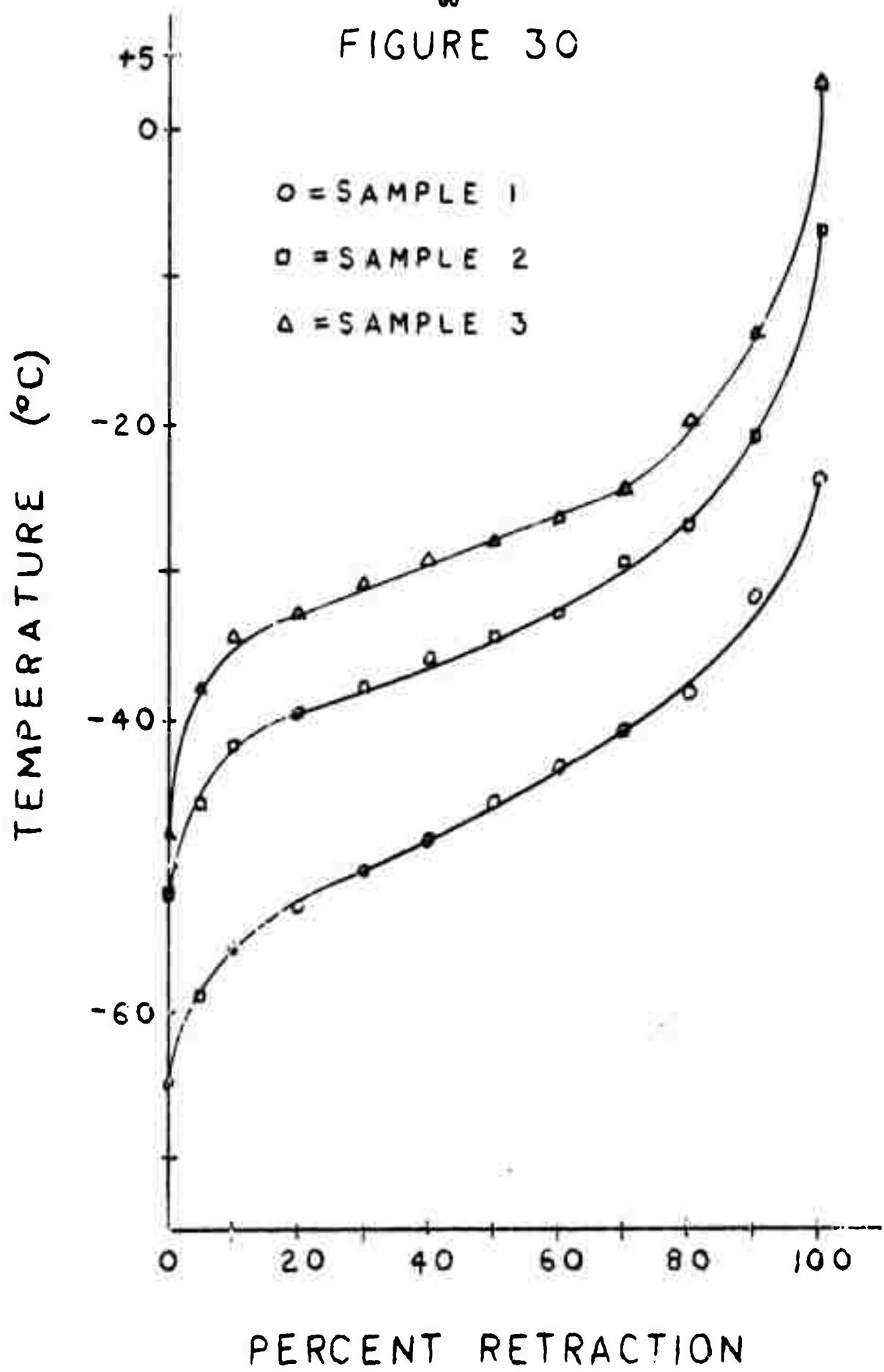
TABLE VIII

Sample Number	1	2	3
Cure Date	1-61	1-54	5-56
TR 10	-55.8°C	-42°C	-34.6°C
TR 30	-50.4	-37.7	-30.8
TR 50	-45.8	-34.7	-28.0
TR 70	-41.0	-29.6	-24.5
(TR 70 - TR 10)	14.8	12.4	10.1
Freezing Point	-59	-44.5	-36

Future Work

Since the difference between the temperature at which a vulcanizate retracts 10 percent (TR 10) and the temperature at which a vulcanizate retracts 70 percent (TR 70) increases as the tendency to crystallize increases, sample no. 1 crystallizes before sample no. 2; and sample no. 2 crystallizes before sample no. 3.

FIGURE 30



The temperature retraction test will be performed on samples that have undergone accelerated aging. It is believed that measurable changes will occur in (TR 70 - TR 10) and hence represent an indication of the degree of aging.

SECTION I

Bibliography

SECTION I

Bibliography

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SECTION XI

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MASS SPECTROMETRY, ELECTRON MICROSCOPE
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